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EMISSION SPECTROSCOPY AND ITS APPLICATION IN THE INVESTIGATION AND SOLUTION OF PROBLEMS IN CERAMICS*

By H. C. Harrison and L. B. Bassett†

Abstract

The fundamental theories of emission spectroscopy and the application of these theories to qualitative and quantitative spectrographic analysis are briefly reviewed. Some of the more widely used analytical methods and techniques are compared as to their relative value in the spectrographic analysis of ceramic raw materials and products. An inexpensive projection enlarger is described.

I. Introduction

The spectrograph in many modified forms has been used in scientific investigation for nearly a century. It has been only within the past thirty years, however, that this highly accurate and time-saving tool has invaded industrial fields. The rapidly growing importance of spectrographic methods has been reflected by the steady increase in the number of scientific papers dealing with the various applications of spectroscopy to science and industry.[1] The phenomenal expansion of the uses of spectroscopy in the metal industries, agriculture, biology, chemistry, medicine, criminology, and ceramics has been due mainly to recent improvements in the construction of instruments, photographic emulsions, and analytical methods.

Spectrographic methods are superior to chemical methods in (1) speed and cost of analysis, (2) sensitivity in the detection and determination of minute quantities of elements, (3) the ability to analyze very small samples, (4) the detection of totally unsuspected elements present in a sample, and (5) for the analysis of substances difficult to analyze chemically, such as refractories, enamels, glasses, slags, ores, rocks, mineral products, cements, and glazes. Spectrographic methods, however, are not as satisfactory as chemical methods for (1) analysis of a majority of the nonmetal elements, such as oxygen, nitrogen, chlorine, or sulfur, (2) certain analyses which can be carried out more quickly and more accurately by chemical or colorimetric methods, and (3) the quantitative determination of elements, in general, that occur in concentrations greater than 10%.

The technique involved in spectrographic analysis is not difficult to acquire, and it is not unusual to assign the taking of the spectrogram to a laboratory assistant, leaving its interpretation to be done at the convenience of the analyst. The identification of the spectrum lines at first appears to be a complicated and time-consuming task, but continued experience will enable the analyst to identify many of the detectable elements by a casual inspection of the spectrogram without accurately determining the wave lengths of the lines; the entire analysis, including the necessary wave-length estimations, can usually be accomplished in a remarkably short time.

The recent increase in the number of publications dealing with the application of spectroscopy to ceramic problems has resulted in numerous requests by ceramists for a survey of the fundamental theories of emission spectroscopy and their application to qualitative and quantitative spectrographic analysis. A detailed knowledge of the construction of spectrographs, the theory of spectral excitation, and the methods employed in emission spectroscopy is not necessarily essential to ceramists who wish to understand published articles or to use spectroscopy in routine analysis. A brief review, however, of this science and its application to the ceramic and allied fields may assist the ceramist who desires to acquire a sufficient knowledge of spectroscopy to enable him to interpret available data and to evaluate spectroscopy and its practical applications to control and research problems.

II. Origin of Spectra

When an atom is excited by a strong electric field, is bombarded by an electron, or is made to collide violently with another atom by means of high temperature, light of definite wave-length values is emitted. The light emission is a result of the movement of one or more electrons in their orbits within the atoms emitting the light. The electrons are displaced to outer orbits by the excitation energy, and as the displaced electrons fall to an inner orbit and eventually to the normal orbit, they liberate this potential energy as monochromatic light. The orbits, or energy levels, in the atoms are sometimes compared to the steps in a flight of stairs, and the movement of an electron from one orbit to another is compared to the movement of an object going upward or downward on the stairs. The object may be placed upward one step at a time, or it may be placed on any higher step without coming to rest on each intervening step on the trip upward. The reverse is true of the downward trip. It should be emphasized that the object cannot come to rest between any two successive steps, that is, it goes up or down by unit steps or multiples of unit steps but never

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[1] (*a*) W. R. Brode, Chemical Spectroscopy, pp. 315–25. John Wiley & Sons, Inc., New York, 1939.

(*b*) W. F. Meggers and B. F. Scribner, Index to Literature on Spectrochemical Analysis, 1920–1937. American Society for Testing Materials, Philadelphia, Pa.

(*c*) G. R. Harrison, Atoms in Action, pp. 155–76. William Morrow & Co., New York, 1939.

by fractions of steps. It is not necessary that the distance between steps or between orbits be a uniform distance or energy state. The energy, E, which is emitted may be expressed as the difference in energy of the two electron states. This is given by the formula,

$$E = W_1 - W_2 = h\nu.$$

Where W_1 = total energy of atom in initial energy state.
W_2 = total energy of atom in final energy state.

This difference in energy is equal to $h\nu$, where ν is the frequency of the emitted light and h is the Planck constant.[2] In practice, wave length, λ, is used instead of frequency and may be obtained by the proper calculation, where c is the velocity of light, $1/\lambda = \nu/c$.

Measurements show that an atom of a given kind always absorbs or emits energy in one or several definite amounts, corresponding to the change of electrons from one energy state to another. The light emitted from a gas or vapor in the incandescent state is the sum of a great number of like transitions, and because each transition corresponds to a monochromatic radiation, the intensity of each monochromatic radiation is proportional to the number of like transitions and this depends on the number of like atoms present in the light source.[3]

The radiations emitted by each element are characteristic of the element, and the spectrum pattern resulting from the separation of these radiations is frequently called the "fingerprint" of the element.

III. Instruments

(1) Spectroscopes and Spectrographs

Spectroscopes and spectrographs[4] are instruments designed to resolve light into its component wave lengths. By means of these instruments, it is possible to analyze a sample both qualitatively and quantitatively for a great number of elements, irrespective of their mode of combination in the sample. The spectroscope and the spectrograph employ the same principles of construction and operation, the former being used for the visual examination of the spectrum and the latter used to record the light radiations as lines on a photographic plate or film. Spectrographs using a glass prism and glass lenses are satisfactory for the examination of the visible and infrared regions of the spectrum but are not satisfactory for work in the ultraviolet region of the spectrum. When the optical parts of a prism spectrograph are composed of quartz or when the prism is replaced by a concave or reflection-type grating, the working range is extended beyond the visible region of the spectrum (4000 to 8000 a.u.) to include the ultraviolet region for the quartz system and to include both the ultraviolet and infrared regions for the grating instruments. The grating produces a spectrum having uniform linear dispersion; the prism produces a spectrum having an abnormal dispersion, that is, the blue end of the spectrum is more widely dispersed than the red end.[5] The usefulness of the spectroscope is limited to the investigation of the spectra of relatively few elements whose strong radiations occur within the visible portion of the spectrum. The spectrograph extends the working range to the ultraviolet and infrared regions of the spectrum, the limits of this range being determined by the type of emulsion on the photographic plate, the composition of the optical parts of the spectrograph, and the absorbing power of air and gelatine.

There is no standard spectrograph for all types of analytical work. The type of instrument used is generally governed by the character of the analysis to be undertaken and by the initial cost of the instrument. At the present time, quartz spectrographs are in wide use, largely because the majority of the elements emit their most sensitive radiations in the ultraviolet portion of the spectrum and quartz transmits the radiations in this range. Grating spectrographs have not been widely used for analytical purposes because of the difficulties encountered in obtaining an adequate supply of gratings and in protecting the grating from fumes and dust encountered under industrial conditions and because the fragile nature of the grating presents a real problem when it is necessary to clean it. Several new models of the grating spectrograph have been placed on the market recently, and it is probable that this type of instrument will come into much wider use because of its low cost, simplicity in construction, and superiority to the prism spectrograph with respect to wider dispersion in the visible and infrared regions of the spectrum.[6]

The size of the spectrograph employed is governed by the character of the elements likely to be encountered during the course of the analysis. The small and medium spectrographs have relatively low dispersion and are suitable for work where elements emitting complex spectra are not encountered or where the instrument is to be used for the study of absorption spectra. Unfortunately, ceramic materials frequently contain elements, particularly iron, which emit complex spectra and therefore a high-dispersion instrument is almost

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[2] N. R. Russell, R. S. Dugan, and J. Q. Stewart, Astronomy, pp. 552–54. Ginn and Co., New York, 1927.

[3] R. C. Johnson, Spectra, pp. 7–8. E. P. Dutton & Co., New York, 1928.

[4] (*a*) W. R. Brode, *loc. cit.*, pp. 27–32.
(*b*) S. J. Lewis, Spectroscopy in Science and Industry, pp. 11–21. Blackie & Son, Ltd., London and Glasgow, 1933; *Ceram. Abs.*, **12** [12] 435 (1933).
(*c*) Bausch and Lomb Optical Co. Catalog, No. D-111, pp. 226–34, 252–59, Jan. 1, 1940.
(*d*) "Grating Spectrogram," *Central Scientific Co. Bull.*, No. 107, p. 1.
(*e*) Spectrographs, Hilger Catalogue E, pp. E1-47, Adam Hilger, Ltd.

[5] G. R. Harrison, "Comparison of Prism and Grating Instruments for Spectrographic Analysis of Materials," *Proc. 5th Spec. Conf.*, pp. 31–37. John Wiley & Sons, Inc., New York, 1938.

[6] (*a*) M. F. Hasler, "Practice of Arc Spectrochemistry with a Grating Spectrograph," *ibid.*, pp. 43–46.
(*b*) W. S. Baird, "Practical Grating Spectrograph for Industrial Use," *Proc. 6th Spec. Conf.*, pp. 80–87. John Wiley & Sons, Inc., New York, 1939.
(*c*) Morris Slavin, "Prism Versus Grating for Spectrochemical Analysis," *Proc. 7th Spec. Conf.*, pp. 51–58. John Wiley & Sons, Inc., New York, 1940.

essential for studying the emission spectra of ceramic raw materials and ceramic products.

(2) Photometers

The measurement of line intensity obtained by the determination of line density is subject to errors of judgment and eye strain when done visually. The true density of lines in a spectrogram is obtained by photometric measurement in accurate quantitative analysis. Instruments used for measuring optical density are modified forms of photometers and are called densitometers, density comparators, microphotometers, and microdensitometers.[7]

IV. Light Sources

The four most commonly used methods for vaporizing and energizing atoms or molecules to emit their characteristic spectra are a nonluminous flame, an arc, a high-tension spark, and a vacuum-tube discharge. Although each method may be used in varied forms, the basic principles of spectral excitation fall under these four classifications with the possible exception of the special case of the explosion of wires.[8]

(1) Flame Sources

Every chemist is familiar with the qualitative test for certain elements which is performed by introducing salts of solutions of these elements into a Bunsen flame and by observing the color of the resulting flame. This method of spectral excitation is useful only for the identification of the alkali metals, the alkaline earths, gallium, thallium, and indium.[9] Lundegardh[10] has modified the flame technique by employing an acetylene-air flame and spraying a solution of the unknown material into the flame. This procedure has been used for the qualitative identification and quantitative estimation of thirty-four of the common elements. The Lundegardh method is especially adaptable to the spectrographic examination of biological and agricultural material and of some minerals.[8]

(2) Arc Sources

The d.-c. arc is the most widely employed and perhaps the most generally useful light source for spectrographic analysis.[11] Its advantages are (1) a relatively high temperature (1000° to 6000°C.); (2) simplicity in construction and operation; (3) applicability for use with samples which may be solid pieces, powders, or solution; (4) the consumption of the entire sample resulting in a more representative analysis of a heterogeneous sample; and (5) its nondependence on the electrical conductivity of the sample. In the case of metals and alloys, the sample may be used as electrodes, but the usual arcing technique is to place the sample, in the form of a powder, in the crater of a carbon, copper, or silver electrode and to strike an arc between the sample and another electrode. The electrode holding the sample may be either the anode or the cathode. When the sample is in solution, it may be analyzed by evaporating portions of the solution on a solid electrode and then arcing the dried sample in the usual manner.

The cathode layer method of analysis developed by Mannkopff and Peter at Göttinger is sensitive, and it is particularly applicable to the analysis of small amounts of nonconducting solids.[12]

The interrupted arc and the high-voltage a.-c. arc are recent modifications which have shown considerable promise.[13]

All of the metals and the nonmetals, arsenic, carbon, boron, phosphorus, and silicon, emit their characteristic spectra in the arc. Fluorine may be detected by the presence of distinctive CaF_2 bands if the sample contains calcium.

(3) Spark Sources

The condensed spark circuit and many modifications of the circuit have been used successfully as a spectroscopic light source.[14] The temperature in such sources is generally higher than that obtained in the flame or arc, and the scope of analyses is widened to include the elements chlorine, bromine, iodine, oxygen, nitrogen, sulfur, selenium, and tellurium. The spark is particularly adaptable to the analysis of low-melting alloys and of liquids; it is also widely used for the analysis of a spot on a larger surface; and it is

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[7] (*a*) W. R. Brode, *loc. cit.*, pp. 91–94.
(*b*) Spectrographic Outfits for Metallurgical and General Chemical Analyses, 8th ed., pp. 52–53. Adam Hilger, Ltd., London, 1936.
(*c*) Bausch and Lomb Optical Co. Catalog, No. D-111, pp. 264–65, Jan. 1, 1940.
(*d*) Walter Gerlach and Eugen Schweitzer, Foundations and Methods of Chemical Analysis by Emission Spectrum, pp. 50–52. Adam Hilger, Ltd., London, 1929; *Ceram. Abs.*, **11** [7] 431 (1932).

[8] F. Twyman, Practice of Spectrum Analysis with Hilger Instruments, 6th ed., pp. 14–16. Adam Hilger, Ltd., London, 1935.

[9] (*a*) W. R. Brode, *loc. cit.*, pp. 44–45; (*b*) *Ibid.*, pp. 83–88.
(*c*) S. J. Lewis, *loc. cit.* p. 23.

[10] H. Lundegardh, Die quantitative Spektralanalyse der Element, Part I. G. Fischer, Jena, 1929; Part II, 1934.

[11] (*a*) W. R. Brode, *loc. cit.*, pp. 45–50.
(*b*) F. Twyman, *loc. cit.*, pp. 16–19.
(*c*) S. J. Lewis, *loc. cit.*, pp. 23–25.
(*d*) D. M. Smith, Metallurgical Analysis by the Spectrograph, Research Monograph No. 2, pp. 24–31. British Non-Ferrous Metals Research Assn., London, 1933; *Ceram. Abs.*, **13** [8] 225 (1934).

[12] (*a*) R. Mannkopff and C. Peters, "Über quantitative Spektralanalyse mit Hilfe der negativen Glimmschicht im Lichtbogen," *Z. Physik*, **70**, 444 (1931).
(*b*) L. W. Strock, Spectrum Analysis with the Carbon Arc Cathode Layer (Glimmschicht), pp. 1–26. Adam Hilger, Ltd., London, 1936; *Ceram. Abs.*, **17** [10] 341 (1938).

[13] (*a*) W. R. Brode, *loc. cit.*, pp. 50–52.
(*b*) Walter Gerlach and Werner Gerlach, Clinical and Pathological Applications of Spectrum Analysis, pp. 10–13. Adam Hilger, Ltd., London, 1934.
(*c*) O. S. Duffendack and K. B. Thomson, "Developments in Quantitative Analysis of Solutions by Spectroscopic Means," *Proc. Amer. Soc. Testing Materials*, **36** [Part II] 301–309 (1936).
(*d*) J. S. Owens, "Helpful Accessories in Practical Spectrographic Analysis," *Proc. 5th Spec. Conf.*, pp. 17–23. John Wiley & Sons, Inc., New York, 1938.

[14] (*a*) W. R. Brode, *loc. cit.*, pp. 52–54.
(*b*) F. Twyman, *loc. cit.*, pp. 20–23.
(*c*) D. M. Smith, *loc. cit.*, pp. 32–39.
(*d*) Walter Gerlach and Eugen Schwietzer, *loc. cit.*, pp. 33–44.

valuable in certain investigations because it does not destroy the sample. A spectrogram taken with the spark is relatively free from the continuous background found in many spectrograms taken with the carbon arc. It is more constant in character than the arc and has, therefore, found considerable favor as the light source for quantitative spectrographic analysis. It is inferior to the arc in sensitivity and for the analysis of nonconducting solids and heterogeneous samples.

(4) Gaseous Discharge Sources

This type of light source is used for the analysis of the rare gases and hydrogen, oxygen, nitrogen, sulfur, and some molecular compounds. A gas or mixture of gases is placed in a discharge tube at low pressure and excited by electrode or electrodeless discharge.[15] A high-voltage spark spectrum of the contents of the tube is produced. This method of spectral excitation is difficult to control within the limits necessary for accurate qualitative or quantitative analysis.

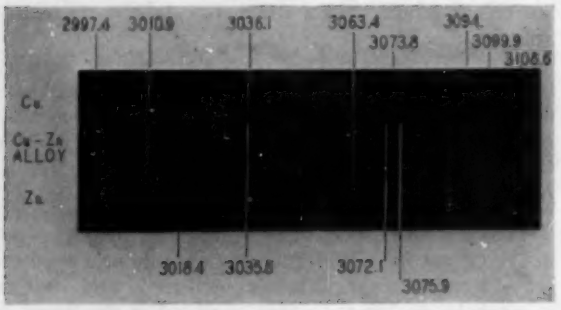

FIG. 1.—Spectrogram of zinc-copper alloy in juxtaposition with metallic copper and zinc.

V. Qualitative Analysis

The lines in the spectrum of the unknown sample may be identified by photographing the spectra of known elements and the spectrum of the unknown sample in juxtaposition for direct comparison or by matching by superposition with lines of known spectrograms taken on separate plates.[16] Figure 1 shows the spectrogram of a copper-zinc alloy photographed in juxtaposition with the spectrograms of metallic copper and metallic zinc. It will be observed that zinc and copper lines appear in the spectrogram of the alloy.

The most accurate method of line identification is to determine the wave lengths of the lines in the spectrogram of the unknown sample. This is most easily accomplished by photographing a comparison spectrum in juxtaposition with the spectrum of the unknown sample, care being taken that the plate is not moved lengthwise during the photographing of the spectra. The comparison spectrum may be produced by the mercury arc, by the mercury-cadmium arc, or by pure iron excited by the arc or spark method. Iron is most frequently used for producing a comparison spectrum because it is inexpensive, easily obtained in a state of high purity, and emits a spectrum containing many lines whose wave lengths are accurately known.

It is rarely necessary to obtain an accurate measurement of wave lengths of all lines in the unknown spectrogram for spectrographic analysis. The identification of several of the strong lines of each element usually suffices for the identification of that element, and this may be accomplished by locating these lines with relation to neighboring iron lines of known wave lengths. Suitable magnification of the spectrograms and the use of prepared charts of the iron spectrum[17] facilitate the determination of wave lengths.

VI. Quantitative Analysis

Quantitative spectrographic analysis is based on the assumption that the number and intensity of spectral radiations emitted by an element under controlled excitation conditions are a measure of the concentration of that element in the light source.

(1) Comparison Methods

The first methods used for quantitative analysis were based on the fact that in a series of samples of known composition in which the concentration of a constituent decreases from member to member, the number of spectrum lines of that constituent decreases proportionally to the concentration of the constituent in the sample.[18] The spectrum lines of the constituent are tabulated in order of their wave length for each different concentration of that constituent, and the number of these lines present in the spectrum of the unknown samples gives an estimate of the concentration of the constituent in the unknown sample. The weight of sample, excitation conditions, and photographic development must be kept constant for the known and unknown spectra.

The so-called comparison methods were the first line intensity methods used. The spectrum of the unknown sample is photographed on the same plate or film with the spectra of a series of standard samples having known but varying compositions. The comparison of line intensity is done visually by the analyst who compares the intensity of certain lines of a chosen constituent in the spectrogram of the unknown sample with the intensity of the same lines in the spectrograms of known composition. A fair degree of accuracy can be obtained by this method, but the procedure is time-consuming owing to the number of spectrograms which must be photographed for each analysis. Wainer and

[15] (*a*) W. R. Brode, *loc. cit.*, pp. 54–58.
(*b*) S. J. Lewis, *loc. cit.*, p. 89.
(*c*) R. C. Johnson, *loc. cit.*, pp. 87–88.
[16] (*a*) W. R. Brode, *loc. cit.*, pp. 61, 72–74.
(*b*) F. Twyman, *loc. cit.*, pp. 31–36.
(*c*) D. M. Smith, *loc. cit.*, pp. 10–17.
(*d*) Walter Gerlach and Eugen Schweitzer, *loc. cit.*, pp. 53–64.
(*e*) W. B. Meldrum and E. T. Glucker, Jr., Introduction to Theoretical Chemistry, pp. 441–42. American Book Co., New York, 1936.

[17] W. R. Brode, *loc. cit.*, pp. 65–70.
[18] (*a*) D. M. Smith, *loc. cit.*, pp. 55–63.
(*b*) W. R. Brode, *loc. cit.*, pp. 77–79.
(*c*) F. Twyman, *loc. cit.*, pp. 37–38.
(*d*) Walter Gerlach and Eugen Schweitzer, *loc. cit.*, pp. 65–67.

Dubois[19] have recently suggested a modification of this method which shows considerable promise for use with ceramic materials.

(2) Logarithmic or Step-Sector Disk Methods

The logarithmic and the step-sector disk methods of quantitative spectrographic analysis[20] depend on the use of a revolving disk placed directly in front of the spectrograph slit. The periphery of the disk may be cut in a smooth logarithmic curve representing an infinite number of steps or it may be cut to have a definite number of steps of equal depth, the angular length of which have a definite ratio from one step to the next in the series. Both sectors provide a means for varying the exposure over the length of the spectrograph slit. Lines produced by the use of these sectors have a graded density on the photographic plate. The logarithmic-sector disk produces a tapered line which is dense at one end and vanishes at the other end. The step-sector disk produces a line which has a definite number of segments graded as to line density and width. Figure 2 shows a spectrogram taken with a step-sector disk. The lines produced by the use of the step-sector disk are often used for plate calibration. Special slits are sometimes used to produce the same effect. The measurement of density is obtained by a measurement of the length of the line on the photographic plate. Calibration must be made with comparable samples containing known percentages of the constituent under investigation.

FIG. 2.—Spectrogram taken with step-sector disk.

(3) Internal Standard Method

The most widely used method of quantitative spectrographic analysis depends on the use of the so-called internal standard.[21] Lines of the constituent to be determined are compared with lines of another constituent present in the same sample in controllable amounts. In many cases, the control lines are lines of the main constituent of the sample, but in some cases it is necessary to add a constituent to all samples in such amounts that each sample contains the same percentage of the added constituent. Care must be taken that the added constituent is not present in any of the original samples, or if it is present, its concentration must be accurately known. Under accurately controlled excitation and photographic conditions, the control lines will have constant densities, but if conditions are not constant, the unknown and control lines should be equally affected, and the logarithm of the ratio of the intensity of these lines should be practically constant.

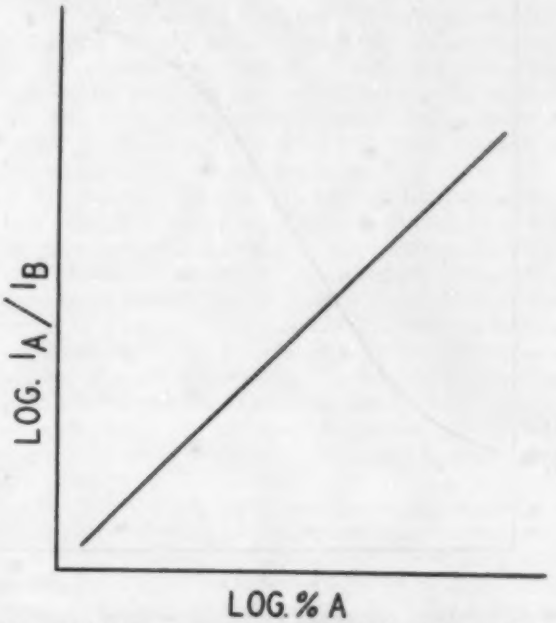

FIG. 3.—Working curve; selected lines of elements A and B are used; I = line intensity.

(4) Working Curve and Plate Calibration

Accurate quantitative spectrographic analysis requires the use of a densitometer or some other modification of a photometer to measure line density or blackening. A working curve must be prepared before the actual analysis[22] is made (Fig. 3). The data used to plot the curve is obtained from spectrograms of a series of accurately prepared standard samples which contain a fixed quantity of the control constituent and varying quantities of the constituent to be determined. The logarithm of the ratio of the intensity of a chosen line of the constituent to the intensity of a chosen line of the control constituent is plotted against the logarithm of the percentage of the constituent to be determined in the sample.

The variation of line intensity in different regions of the spectrum, that is, density, is not a linear function of the intensity of the light, and nonuniformity of photographic characteristics of emulsions necessitates

[19] Eugene Wainer and E. M. Dubois, "Spectroscopic Analysis of Enamel Frits," *Bull. Amer. Ceram. Soc.*, 20 [1] 4–7 (1941).

[20] (*a*) W. R. Brode, *loc. cit.*, pp. 83–88.
(*b*) F. Twyman, *loc. cit.*, pp. 40–42.
(*c*) D. M. Smith, *loc. cit.*, pp. 48–51.

[21] (*a*) W. R. Brode, *loc. cit.*, pp. 80–81.
(*b*) D. M. Smith, *loc. cit.*, pp. 45–48.
(*c*) Walter Gerlach and Eugen Schweitzer, *loc. cit.*, pp. 67–74.

[22] (*a*) W. R. Brode, *loc. cit.*, pp. 88–89.
(*b*) L. W. Strock, *loc. cit.*, pp. 28–29.

plate calibration for increased accuracy.[23] The data used for plotting this curve were obtained from a spectrogram taken with the use of a step-sector disk or its equivalent and a constant light source or from the spectrogram being analyzed, if a step-sector disk is used when taking the spectrogram. Density or blackening of the emulsion is plotted against the logarithm of the light intensity producing the blackening, and the resulting curve is used to convert densitometer readings into line intensity. Only the straight-line portion of the calibration curve can be used for accurate analysis. Figure 4 shows the plate calibration curve.

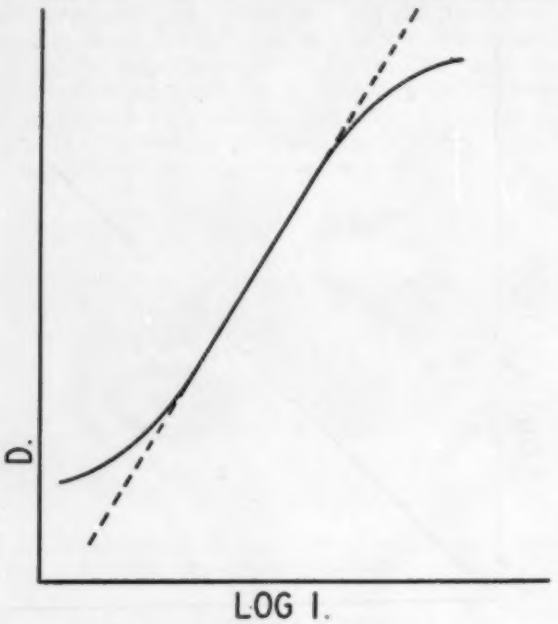

FIG. 4.—Plate calibration curve; I = light intensity, D = density.

VII. Applications of Spectroscopy

(1) Mineralogy

The mineralogist has used spectroscopy for (1) determining the qualitative and quantitative composition of minerals; (2) rapid determination of confusing common minerals; (3) study of different varieties of the same mineral differing only in color; (4) identification of certain gem stones; and (5) detection and quantitative determination of small quantities of boron in minerals.

A knowledge of the chemical composition of an unknown sample is of great aid to petrographic identification. If the petrographer is acquainted with the qualitative and has a rough estimation of the quantitative composition of a sample, he can usually eliminate at once all but a small group of possibilities. A knowledge of the presence or absence of trace elements also often proves to be a valuable clue in differentiating between several closely related substances.

[23] (*a*) W. R. Brode, *loc. cit.*, pp. 90–91.
(*b*) L. W. Strock, *loc. cit.*, pp. 29–33.

(2) Glass Technology

Glass technologists have employed spectrographic analysis for (1) rapid qualitative analysis as a basis for planning chemical analysis; (2) testing the effectiveness of chemical separations during the course of the chemical analysis; (3) testing the purity of glass batches; (4) quantitative determination of minor constituents; (5) an aid in the identification of glass inclusions; and (6) study of spectral transmission of glass.

(3) Ceramics

The ceramist has used the spectrograph (1) to check raw materials to see if they meet required specifications; (2) to test for the presence of fluorine and to determine it quantitatively; (3) for rapid qualitative and quantitative analyses of enamels; (4) to differentiate between the alkali metals when they are present in ceramic materials; (5) to study the effect of temperature on volatility of glaze and enamel constituents; (6) to study the effect of impurities on the electrical conductivity of ceramic bodies; (7) to discover the cause of discoloration of brick, tile, pottery, sanitary ware, and other ceramic products; and (8) to examine and classify fragments of ancient pottery.

Ceramic products cover diversified fields, and consequently the spectroscopist is often faced with the necessity of analyzing a heterogeneous assortment of samples. During the past year, samples of the following materials have been submitted to the New York State College of Ceramics for spectrographic analysis:

(1) Several different types of abrasives to determine chemical composition, type of bond employed, and reasons for nonuniformity of color.

(2) Numerous samples of cements to determine chemical composition and to classify as to type.

(3) Electrical insulators for the identification of intrusions and blebs.

(4) Commercial enamels for the identification of fluxes, pigments, and opacifiers.

(5) Dental enamel to determine chemical composition.

(6) Various samples of glass to analyze for general composition, for trace elements, to identify inclusions, and to use for general classification.

(7) Glaze samples to determine chemical composition and to study behavior during firing with special reference to volatile constituents.

(8) Several different types of refractories for identification of chemical constituents; for the determination of the type of bond used; for the analysis of protective coatings; and for the identification of elements occurring as impurities.

(9) Plugs used in steel furnaces.

(10) Roofing for the identification of fluxes and color intensifiers.

(11) Pieces of sanitary ware to determine cause of blemishes.

(12) Sewer pipe to determine fluxes, materials used in glazing, and reasons for abnormal color effects.

(13) Siding to classify cement, pigments, and fluxes and to analyze coating.

(14) Numerous samples of pottery to determine glaze composition and the cause of blemishes.

(15) Pieces of ancient pottery to determine method of obtaining unusual decorative effects.

(16) Several clays to find relationship between reheat expansion and chemical composition.

(17) Samples of feldspar to analyze for small traces of unusual impurities and the alkali metals, Li, Rb, and Cs.

The spectrographic analysis did not solve all of the problems submitted, but in a majority of cases the analysis was of definite value in the ultimate solution of many problems. Many of the difficulties were due to the presence of totally unsuspected elements or compounds, and increasing evidence supports the contention that a more thorough knowledge of the part played by trace elements is important to the ceramist.

VIII. Experimental and Analytical Difficulties

(1) Heterogeneous Material

The greatest difficulty encountered in the spectrographic analysis of ceramic raw materials or fired articles is to obtain a representative sample. Ceramic materials usually are not homogeneous, and when the material to be sampled is a fired article, the crushing and grinding operations are sources of contamination. When several samples are fired to completion, using a rotating sector disk to avoid excessive background intensity, the sampling errors are partially compensated.

(2) Obtaining Samples of Glazes

The sampling of a blemish on the surface of a glazed body is frequently encountered. Glaze usually adheres tenaciously to the body, and it is almost impossible to obtain a sample without including some body material. The procedure to follow in this case is to take spectrograms of equal weights of the body, discolored glaze, and glaze, and the elements not common to all or in greater concentration in one may be compared to reveal the offending material.

(3) Complexity of Spectra

The spectra of ceramic materials range from the simplest to the most complex likely to be encountered anywhere. The elements encountered include most of the periodic table. For this reason, low-dispersion instruments, such as the small or medium spectrographs, are of value in only a limited number of cases. A large quartz spectrograph or a grating spectrograph are satisfactory instruments for work in that part of the spectrum below 4000 a.u. The principal lines of the alkalis, alkaline earths, most rare earths, the refractory metals of the tungsten group, and several of the common heavy metals unfortunately occur in the visible and red end of the spectrum. If a grating is used or if the quartz optical parts of the spectrograph are replaced by flint glass, the instrument becomes suitable for analysis in the visible and near infrared region of the spectrum. Several spectroscopists advocate the use of the grating spectrograph throughout the spectrum.[14]

IX. Comparison of Analytical Methods and Techniques

(1) Excitation Methods

Comparative studies have been made of the following methods for producing the spectra of ceramic materials: (1) condensed spark; (2) 2000-volt a.-c. arc; (3) d.-c. arc with anode excitation; (4) d.-c. arc with cathode excitation; and (5) d.-c. arc using fractionating electrodes and anode and cathode excitation.

The spectrograms obtained were examined for relative sensitivity and ease of reading. The conclusions were as follows:

(1) The condensed spark is not entirely satisfactory when the sample is heterogeneous, nonconducting, or of a refractory nature. When nonconducting samples are used, the arc tends to jump to the walls of the electrode and the sample is usually blown from the crater of the electrode. This excitation method is less sensitive than d.-c. arc excitation.

(2) Because the a.-c. arc has not been submitted to a conclusive test at this laboratory, judgment will not be passed as to its value in the spectrographic analysis of ceramic materials. Preliminary investigations, however, indicate that because of the difficulties encountered in attempting to retain the powdered sample on the electrode, the d.-c. arc is more satisfactory as a method of spectral excitation. It is admitted that the a.-c. arc has the advantages of ease of maintenance, extreme sensitivity, and low background density, but it is dangerous to use unless special safety precautions are taken.

(3) The d.-c. arc using anode excitation is the most satisfactory method for the spectrographic examination of material containing refractory constituents, and it is as sensitive as the cathode layer method when the amount of sample is not limited. It is convenient to use and is of particular value when a refractory sample is to be fired to completion. In this method, the sample is placed on the anode.

(4) The d.-c. arc, using cathode excitation and focusing the middle portion of the arc column on the slit of the spectrograph, is used in a few special cases where it is necessary to volatilize the sample slowly. When the amount of sample is small, as in the electrode blanks, there is a marked gain in sensitivity in the cathode layer method,[12] wherein the sample is vaporized from the cathode and the light from the arc column immediately above the cathode is focused on the slit of the spectrograph. The increased sensitivity is due to an enrichment of the elements in this region of the cathode.

(2) Electrodes

Electrodes prepared from commercial Acheson graphite rods are used for most analyses. The electrodes may be purified by any one of several methods, de-

[14] (*a*) See footnote reference 4 (*d*), p. 214.
(*b*) G. R. Harrison, see p. 36 of footnote reference 5.
(*c*) Morris Slavin, see pp. 56–58 of footnote reference 6 (*c*), p. 214.

pending on the apparatus available,[25] or purified graphite electrodes may be obtained from several different supply houses. Copper or silver electrodes are used for the analysis of elements occurring as impurities in graphite electrodes. Three sizes of electrodes are commonly used, namely, graphite rods having diameters of $^3/_{16}$, $^4/_{16}$, and $^5/_{16}$ inch. The electrode craters vary from 5 to 15 mm. in depth, depending on the type of sample. The upper electrode is usually a $^3/_{16}$-in. rod, one end of which is pointed in a pencil sharpener. Either the $^3/_{16}$-in. or the $^4/_{16}$-in. rod is used as the lower electrode, depending on whether the sample is readily volatile or of a refractory nature. It has been found that the $^3/_{16}$-in. rod is better for refractory samples. The crater wall is made as thin as possible to reduce the amount of carbon that is vaporized during the arcing of the sample. A cutting tool may be used to assure regulated depth and a flat top to the electrode.

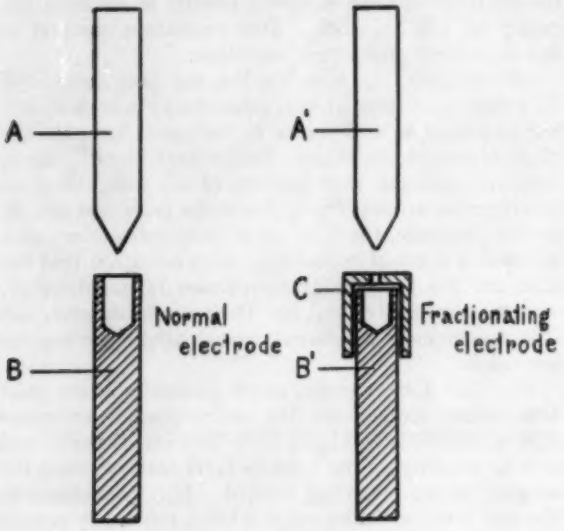

FIG. 5.

An unusual method for analyzing samples that contain very volatile constituents has recently been suggested, based on the use of the so-called boiler or fractionating electrode.[26] When ordinary electrodes are used, it is difficult to keep the more volatile constituents of the sample in the arc stream, with the result that small traces of these constituents are not always detected. When the fractionating electrodes are used, the volatilization of the sample is slower and the vapors are forced in a concentrated stream through the arc and a greater number of atoms are excited to luminosity. Not only does this method show remarkable sensitivity for the detection of this type of element but spectrograms taken with these electrodes have very low background density and are usually free of the spectra of the refractory elements. The fractionating electrode is made in two parts as shown in Fig. 5. Part B is a carbon rod cupped in the usual manner to hold the sample; part C is a cap made from a carbon rod of slightly larger diameter and drilled to fit snugly over part A. A small hole is drilled through this cap. The upper electrode is usually a $^3/_{16}$-in. rod. When the arc is struck, the sample is gradually heated and the constituents appear in the arc stream in the order of their volatility. By proper control of arcing conditions and time of exposure, it is possible to separate the more volatile constituents from the more refractory matrix. The spectrogram obtained shows only the lines of the more volatile elements and the lines of the electrode impurities. Inasmuch as the rate of heating is slower and the vapors must pass through the small orifice into the arc, it is also possible to concentrate the vapors in the arc stream and to decrease the loss of vapor by diffusion and air currents, which results in a more sensitive method for the detection of readily volatile elements.

(3) Special Apparatus and Techniques

A mechanism for moving the plateholder vertically at a constant rate during the photographing of the spectra is used to produce a moving-plate spectrogram, which is of great value for determining the best conditions of arcing and of the exposure time to insure consumption of a representative sample without excessive background.[27] A moving-plate spectrogram should be taken whenever the apparatus is available. The alternate procedure is to take two spectrograms of each sample as follows: (1) with a low-current arc with arcing time so adjusted as to vaporize about one fourth to one half of the total sample placed on the electrode and (2) on another portion of the same plate with high-current arcing of the sample so arranged that the entire sample is vaporized while the spectrogram is being made. These two spectrograms give much the same information as that obtained by the moving-plate procedure.

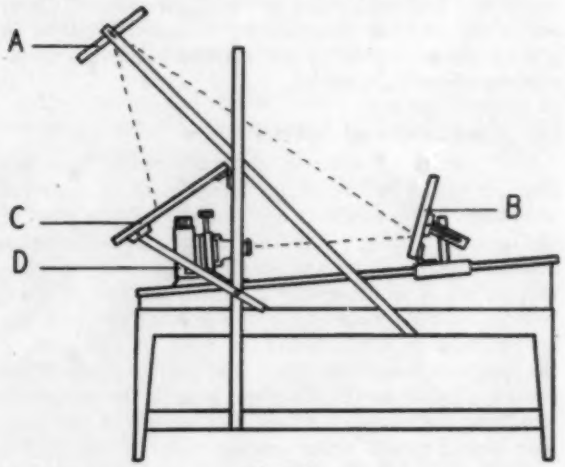

FIG. 6.

[25] J. S. Owens, see pp. 17–18, of footnote reference 13 (*d*), p. 215.
[26] S. S. Cole, personal communication.
[27] (*a*) W. S. Baird, *loc. cit.*, p. 83.
(*b*) David Richardson, "Boiling-Point Determinations by Spectroscopic Methods," *Proc. 5th Spec. Conf.*, pp. 64–70. John Wiley & Sons, Inc., New York, 1938.

Certain samples are found to sputter from the crater of the electrode when the arc is struck. This loss may be avoided by mixing the sample with an equal weight of carbon[28] or by placing 5 to 10 mg. of anhydrous sodium carbonate in the crater above the sample to form a flux when the arc is struck.[29]

An enlarged image of the spectrogram greatly facilitates the identification of the spectrum lines and is an essential feature of many of the instruments used to measure the line density. An inexpensive projection apparatus for use in qualitative spectrographic analysis is shown in Fig. 6. This is built around an Argus projector, *D*, with a removable plateholder placed between the film-holding plates and made with a rack along one edge. A pinion is mounted on the side of the projector so that the plate may be moved to show any desired portion of the 10-in. plate. The projected image is reflected by two mirrors, *A* and *B*, and is impinged on an adjustable screen, *C*, directly in front of the operator. The magnification may be regulated by moving the first mirror and readjusting the tilt of both mirrors to bring the image on the screen. Standard enlargements may be used with this projector, and the method described in Brode's book on chemical spectroscopy has been applied satisfactorily for student use.[30]

[28] E. K. Jaycox and A. E. Ruehle, "Quantitative Spectrochemical Analysis of Alloys, Solutions, and Powders," *Proc. 7th Spec. Conf.*, pp. 12–13. John Wiley & Sons, Inc., New York, 1940.

[29] W. C. Pierce, O. R. Torres, and W. W. Marshall, "Qualitative Spectrographic Analysis in the Arc with Graphite Electrodes," *Ind. Eng. Chem., Anal. Ed.*, **12**, 43 (1940).

[30] W. R. Brode, *loc. cit.*, pp. 71–73.

X. Summary

The d.-c. arc using anode excitation is the most generally useful light source for the spectrographic examination of ceramic raw materials and products. Readily volatile constituents of a sample are more easily detected with the use of fractionating electrodes. Because a great many ceramic materials emit complex spectra, the use of a high dispersion spectrograph is recommended for the spectrographic analysis of this class of materials. The moving-plate technique and the projection of an enlarged image of the spectrogram greatly facilitates spectrographic analysis.

Acknowledgment

The authors are deeply indebted to M. E. Holmes and the other members of the faculty of the New York State College of Ceramics for their many helpful suggestions.

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PHOSPHATES IN CERAMIC WARE: I, IN OPAL GLASSES*

By Woldemar A. Weyl

Abstract

The theory has been advanced that the formation of apatite crystals is responsible for opacity in bone-ash opals, which agrees with the experience of the writers in the formation of this group of minerals in magmatic melts. It also explains the characteristic behavior of bone-ash glasses and provides means to avoid certain defects. The importance of phosphate opacification for opal glasses resistant to heat shock is briefly discussed.

I. Introduction

Phosphates have penetrated nearly all of the different fields of ceramics. They are used (1) in refractories to accelerate the inversion of quartz into cristobalite and tridymite as well as to prevent the formation of the stable phase in calcium orthosilicates; (2) in glazes as the chief constituent; (3) in enamels, chiefly in countries which have no domestic sources of boric acid; and (4) in glasses, they are used (*a*) in phosphate crowns for the removal of the secondary spectrum; (*b*) in colorless heat-absorbing types as the essential ingredient; and (*c*) in fluorescent tubing for the development of cold light sources. From among these applications, however, only two uses of phosphates are well known to the ceramist, namely, in bone-ash opal glass and in bone chinaware.

It is the aim of the first two papers of this series to review the facts that are known and to offer a possible explanation of the phenomena in these types of ceramic products.

II. Phosphate Opals Versus Fluoride Opals

The use of bone ash as an opacifying agent for making milk glasses dates far back into the history of glass. In his paper on the history of phosphate glasses, Ganzenmüller[1] mentions two different formulas used by Italian glassmakers of the 14th and 15th centuries. Neri[2] makes no mention of it in his famous book, and he describes only tin oxide as a means of producing milky glass. In Bohemia and Silesia, bone-ash opals have been manufactured on a commercial scale since 1670, and there are formulas of bone glasses made in

* Presented at the Forty-Third Annual Meeting, The American Ceramic Society, Baltimore, Md., April 1, 1941 (Glass Division). Received February 3, 1941.

[1] W. Ganzenmüller, "History of Phosphate Glasses," *Glashütte*, **68** [51] 873–75 (1938); *Ceram. Abs.*, **18** [7] 178 (1939).

[2] Antonio Neri, De Arte Vetraria.

the year 1796 in Bohemian glass plants which are still in existence. As Ganzenmüller pointed out, the revival of interest in phosphate opals in the second half of the 17th century is due to the general trend of finding new ways to imitate the oriental chinaware. The Réaumur porcelain, a highly devitrified glass, offers an excellent example. The addition of bones, fresh or calcined, therefore, has probably been considered as another way to change glass into porcelain.

Bone ash and, later, guano were the chief sources of phosphates for a long time. When their value as fertilizers was recognized, however, the demand for them increased rapidly and, as a consequence, the prices increased and phosphates became too expensive for the glassmaker. Other opacifiers were needed, and around 1860 the fluoride opal glasses were discovered in the Philadelphia area. This "hot cast porcelain," as it was called, soon dominated the market because the use of cryolite was not only less expensive but the fluoride opal glasses were much easier to melt and they did not show certain defects which were characteristic of bone-ash glasses. In 1885, the Oeresund Company[3] monopolized the cryolite deposits which were in the hands of the Danish government. The price of cryolite increased, and as a result the interest in bone-ash opacification revived.

Bone ash, used as an opacifier, offered a way to make opal glasses resistant to heat shock.[4] Interest in this process remained even after it was learned how to make fluoride opal glasses, not only with fluorspar but also with sodium silicofluoride and some source of alumina. When Schott and his co-workers discovered the heat-resisting glasses of the borosilicate type, they found that these glasses could not be opacified with cryolite in the usual way, and since then numerous attempts have been made to control the properties of bone-ash glasses on a borosilicate basis.

III. Nature of Bone-Ash Opacification

Some authors state that phosphates are insoluble in silicate glasses and therefore give rise to immiscibility, which causes a second glassy phase to appear during cooling; the composition of this phase, however, is unknown. According to this explanation, which seems to have been generally accepted, phosphate opacification would belong in the same group as certain lead-borate glasses which can form opaque masses because of immiscibility.

Rooksby[5] recently studied the opacification of lead glasses by means of X rays. His chief interest was the effect of arsenic, but he also made valuable contributions to the nature of phosphate opacification. In lead glasses, he found the second phase to be lead arsenate. When he replaced arsenic by phosphorus, an opal glass resulted in which lead phosphate could be identified. In earlier work on this subject, Fuwa[6] attributed the opacification to the precipitation of phosphorus trioxide. This, however, would be in contradiction to the chemistry of this compound.

Most magmas contain a certain amount of phosphoric acid. Despite the relatively low concentration, which rarely exceeds 0.5%, some phosphate minerals belong to the primary crystallizing phases. Their euhedral forms, indicating that their crystallization has ended, reveal that they have a very low solubility in silicate melts. The phosphorus usually forms minerals belonging to the apatite group which is comprised of many different compositions.

(1) Apatite Minerals

The composition of apatite is either $3Ca_3P_2O_8 \cdot CaF_2$ (fluorapatite) or $3Ca_3P_2O_8 \cdot CaCl_2$ (chlorapatite); in addition, there are intermediate compounds containing both fluorine and chlorine. According to Zambonini,[7] the ratio of calcium bound to phosphorus trioxide and of calcium bound to fluorine may vary considerably. Some apatites contain the hydroxy group instead of fluorine; others show partial replacement of calcium by manganese or even by the rare oxides. Synthetic experiments, which have supplemented the earlier conclusions based only on rock analysis, have increased the knowledge of this interesting group of minerals. Of particular interest is the fact that the group of apatites is isomorphous with the group of pyromorphites and mimetites[8]; the former (pyromorphites) is derived from the phosphate of lead with lead fluoride or chloride, and in the mimetites, arsenic replaces the phosphorus. Whenever possible, a mineral of one of these groups will form. The crystal lattice of the apatite is found in the animal world, forming the basic substance of bones and teeth, in the form of hydroxyapatite and fluorapatite. In the laboratory, the formation of apatite is observed as a reaction product, whereas pure phosphates would be expected. This tendency is so pronounced that, in 1908, Basset[9] expressed doubt that a chemist had ever handled a pure calcium phosphate corresponding to the formula $Ca_3P_2O_8$. By analysis of the phosphates in trade, all deviate more or less from this theoretical composition.

The technologist makes use of the strong tendency to form apatite by the removal of fluorine from potable water. Investigations carried on by MacIntire and Hammond[10] as well as by Adler and Klein[11] showed that traces of fluorine can be removed from drinking

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[3] C. Weinreb, "Cryolite Glass," *Dingler's Polytech. Jour.*, **256**, 361–67 (1885).

[4] E. Zschimmer, K. Hesse, and L. Stoess, "Roughening of Calcium Phosphate Glasses for Lighting Purposes," *Sprechsaal*, **58**, 513–17, 529–34 (1925); *Ceram. Abs.*, **5** [12] 392 (1926).

[5] H. P. Rooksby, "Lead Arsenate and Lead Phosphate in Opal Glasses," *Jour. Soc. Glass Tech.*, **23**, 76–81 (1939).

[6] Kitsuzo Fuwa, "Coloring Glass with Phosphates," *Jour. Japan. Ceram. Assn.*, **32** [382] 431–40 (1924); *Ceram. Abs.*, **5** [1] 18 (1926).

[7] F. Zambonini, "Some Observations on Composition of Apatites," *Compt. rend.*, **162**, 919–21 (1916).

[8] M. Amadori, "Research on Group of Pyromorphite Minerals," *Gazz. chim. ital.*, **49** [I] 38–102 (1919).

[9] H. Basset, "Contributions to Study of Calcium Phosphates," *Z. anorg. Chem.*, **59**, 1–55 (1908).

[10] W. H. MacIntire and J. W. Hammond, "Removal of Fluorides from Natural Waters by Calcium Phosphates," *Ind. Eng. Chem.*, **30** [2] 160–62 (1938).

[11] Howard Adler and George Klein, "Removal of Fluorides from Potable Water by Tricalcium Phosphate," *ibid.*, **30** [2] 163–65 (1938).

water by allowing the water to flow over calcium phosphate, or, more correctly, hydroxyapatite. Under these conditions, fluorapatite, a highly insoluble phase, is formed.

(2) Formation of Apatite in Phosphate Opal Glasses

Based on the knowledge that apatites form whenever possible, the explanation is suggested that these crystals are also responsible for phosphate opacification. No particular effort has been made until now to identify the phase precipitated in soda-lime-silica glasses when bone ash is used as an opacifier. From the work of Zschimmer, Hesse, and Stoess,[4] a series of photomicrographs is available which shows some unusually large crystals formed in commercial bone-ash glasses. Their hexagonal forms as well as their low birefringence agree perfectly with their nature as apatite minerals. The same authors describe the formation of the opaque phase, and they state that the first appearance of opacity is accompanied by small spherulitic crystals resembling little droplets, which are probably responsible for the picture of immiscibility as the reason for opacification. The composition of the apatite crystals may be expressed by the general formula,

$$3M_3R_2O_8 \cdot MX_2.$$

In which M = Ca, Pb, Mn, and others.
R = P, As, or V.
X = F, Cl, or OH.

For the purpose of the present studies, the group of vanadates may be completely excluded. The participation of arsenic, however, seems probable, especially in certain types of glasses where arsenic has been introduced as a fining agent. Riedel[12] noticed that opacification occurred in soda-lime-silica glasses when 3% of arsenic was added and the oxidizing atmosphere had caused most of it to change into the pentavalent state. Rooksby[5] discussed the crystal structures of lead arsenate and lead phosphate formed in opal glasses and their relationship to the group of apatite minerals. Although it is not yet known how far arsenic can be replaced by antimony, the very fact that fluorine and calcium both play an important role in opacifying enamels with antimony compounds seems to indicate that here too a similar relationship exists.

The role of fluorine can be readily demonstrated by attempting to make opals from pure phosphates. Only inhomogeneous and cloudy glasses are obtained. The addition of small amounts of sodium fluoride or fluorspar, however, changes these glasses into distinct opals. In using bone ash as a raw material, a certain amount of fluorine is always introduced with the phosphate. The effect of additional fluorine, therefore, is not very pronounced in true bone-ash glasses. When pure antimonates are used in enamels for opacification, fluorine has a striking effect on the density of opacification. The amounts of fluorine under discussion are in no way sufficient to cause opacity by themselves.

From this experience, group MX_2 is shown to be of particular interest and importance. Replacing fluorine by chlorine probably would not offer any advantage because the introduction of chlorides into high melting glasses results in several technical difficulties. Under certain conditions, however, hydroxyapatite may form. The French mineralogist, Damour,[13] as early as 1856 described the replacement of fluorine by hydroxy groups in certain apatites. His findings have been confirmed, and in recent years Körber and Trömel[14] have pointed out that in the system, CaO–P_2O_5, hydroxyapatite forms at temperatures as high as 1050°C. whenever moisture is not carefully excluded. According to Schleede, Schmidt, and Kindt,[15] hydroxyapatite starts to lose water at 1400°C., which causes the lattice to break down completely.

IV. Roughness and Brittleness as Chief Defects of Phosphate Glasses

If there is insufficient fluorine, chlorine, or water present in the glass, the formation of apatite crystals in the glass obviously cannot take place. Under these circumstances, a phenomenon can be observed which is considered to be characteristic of phosphate glasses. During shaping, particularly blowing, the glass comes in contact with the moist atmosphere and a reaction starts at its surface which leads to the formation of hydroxyapatite. As these crystals grow out of the otherwise smooth surface, they cause the glass to become rough. This roughness has been considered to be the chief difficulty in manufacturing phosphate opals and attempts have been made to correct it. Zschimmer[4] and his co-workers state that Schott and Zsigmondy have already made substantial efforts to overcome this difficulty which has proved to be a serious handicap in making heat shock-resisting opals. Zschimmer studied the effect of bone ash on glasses of the following compositions, with particular attention to workability and roughness:

	(1)	(2)
SiO_2	66.4	60.4
Al_2O_3		6.0
B_2O_3	23.1	23.1
Na_2O	10.5	10.5

The lack of sufficient divalent ions, such as calcium or lead, prevents their opacification by fluorine. The addition of 4 to 8% of bone ash, however, gives a satisfying opacity. The composition of the bone ash used for these experiments was tricalcium phosphate 84, calcium carbonate 10, calcium fluoride 4, and magnesium phosphate 2%.

From this analysis, bone ash is found to contain a substantial amount of fluoride. The amount, however, is not sufficient to prevent the formation of hydroxyapatite because a certain portion of fluorine remains

[12] L. Riedel, "Use of Arsenic for Opacifying Alkali-Lime-Silicate Glasses With or Without Alumina," *Sprechsaal*, **59**, 237 (1926); *Ceram. Abs.*, **5** [9] 274 (1926).

[13] A. Damour, "The Mineral Hydroxyapatite," *Ann. mines*, **10**, 65–68 (1856).

[14] F. Körber and G. Trömel, "Investigations of Lime-Phosphoric Acid Compounds and Lime-Phosphoric Acid-Silica Compounds," *Z. Elektrochem.*, **38**, 578–82 (1932).

[15] A. Schleede, W. Schmidt, and H. Kindt, "Calcium Phosphates and Apatites," *ibid.*, **38**, 633–41 (1932).

in the glassy phase and does not have a chance to participate in the formation of apatite.

The effect of bone ash in soda-lime glasses has been studied by Inwald,[16] who determined the influence of glass composition on meltability and on the extent of opacity. Fuwa[6] introduced phosphates into two series of glasses which had the general formulas of $K_2O \cdot RO \cdot 3SiO_2$ and $K_2O \cdot RO \cdot O, 5B_2O_3 \cdot 3SiO_2$, wherein RO represented the oxides of barium, calcium, zinc, or lead.

In this connection, the work of Knudsen[17] on dispersely colored glass should also be mentioned. The term, "dispersely colored," was applied to those glasses whose color did not originate from selective absorption of light but from selective dispersion of light. The dispersion curves, that is, the refractive indices plotted against wave length, of the two phases intersected within the visible region. Light of this particular wave length, having the same refractive index in both phases, is transmitted without appreciable loss of intensity. Light of any other wave length, however, is scattered, with more dispersion occurring with an increasing difference in the refractive indices of the two phases. Knudsen studied the color change of different glasses in the system Na_2O–PbO–SiO_2–$Ca_3P_2O_8$ when different oxides, such as titanium dioxide, antimony oxide, and thallium oxide, were added. These glasses, however, are of such a complicated composition that it is impossible to draw conclusions on the nature of the second phase. Crystals might be expected of the apatite and pyromorphite group as well as true immiscibility.

(1) How to Decrease Roughness and Brittleness

The roughness of bone-ash opals, as far as it is due to the reaction of the glass surface with the moisture of the atmosphere, can be avoided by two means. (1) It is essential that all constituents necessary for the formation of the apatite are incorporated in the glass proper so that it loses its reactivity with the moisture. (2) Bone-ash opals, however, are characterized not only by their surface roughness but also by their tendency to become brittle because of the large size of the crystals formed. In order to check this defect, it is necessary to find means of avoiding extreme crystal growth. Ways of remedying these characteristic defects are discussed.

The crystal structure of the apatite is sufficiently known from the work of St. Náray-Szabó[18] and Mehmel.[19] Both authors, working independently, arrived at the same conclusion. The position of the fluorine atom within the lattice is such that sufficient space is available for replacing fluorine not only by OH groups but even by the larger chlorine without materially changing the lattice parameters. The amount of chlorine or water which can be present in high melting glasses is limited so that, from this point of view, fluorapatite must still be considered as the chief source of opacity. Assuming roughly that 5% of this phase is necessary to produce the opacity required, a simple calculation shows that such a glass theoretically would need not more than 0.2% of fluorine to change the phosphate into an apatite. As has been pointed out before, all of the fluorine cannot be expected to enter the apatite phase and the glass. A substantially higher amount therefore should be introduced. Only a sufficient amount of fluorine present in the glass will prevent further reaction with the atmosphere as well as the development of surface roughness.

A great deal of the roughness and brittleness in bone-ash glass is due to the size of the crystals formed. The growth of crystals is known to be primarily a function of the viscosity of the glass. Apatites which have high melting points and small solubilities in the silicate melts tend to crystallize out in a temperature range where the viscosity is still very low. Rapid diffusion and the formation of large crystals are the result. An increase in the viscosity of the glass in the crystallization range would necessarily impair its melting qualities. Thus any attack on the problem from this angle cannot be considered to be a practical remedy. Earlier workers in this field have pointed out that some improvement of the quality can be obtained by choosing the proper cooling schedule, but the possible changes here are limited and not too much can be expected from this practice.

The wide range of compositions of the apatite phase proper fortunately offers other possibilities. In substituting lead oxide for calcium oxide, a change is made from the apatite group of minerals to the isomorphous group of pyromorphites, which have a considerably lower melting point. According to Nacken,[20] the melting point of fluorapatite is around 1650°C. and that of chlorapatite, 1530°C. The melting points of the corresponding lead compounds have been determined by Amadori,[8] who synthesized the various pyromorphites and determined the phase equilibria of the lead phosphate, arsenate, and vanadate with lead fluoride and chloride. Amadori states that when calcium is replaced by lead, the melting points of fluorapatite and of chlorapatite are lowered to 1098° and 1156°C., respectively. A further lowering of the melting point may be obtained by progressing from the pyromorphite group to the mimetite group by substituting arsenic for phosphorus. Unfortunately, no corresponding antimony compounds have been studied, and those occurring in nature are not members of the apatite family.

V. Conclusions

From purely mineralogical considerations, it is concluded that a partial replacement of calcium oxide by lead oxide should have beneficial effects on the proper-

[16] O. Inwald, "Studies of Composition and Properties of Phosphate Glasses," *Sprechsaal*, **33**, 1498–99, 1533–34, 1564–65, 1596–97 (1900).

[17] Erik Knudsen, "Dispersely Colored Two-Phase Glasses," *Kolloid-Z.*, **69** [1] 35–43 (1934); *Ceram. Abs.*, **15** [7] 202 (1936).

[18] St. Náray-Szabó, "Structure of Apatite," *Z. Krist.*, **75**, 387–98 (1930).

[19] (*a*) M. Mehmel, "Structure of Apatite," *ibid.*, **75**, 323–31 (1930).

(*b*) M. Mehmel, "Relation Between Crystal Structure and Chemical Formula of Apatite," *Z. physik. Chem.*, **B15**, 223–41 (1931).

[20] F. Nacken, "Formation of Apatite," *Zentr. Mineral., Geol.*, 545 (1912).

ties of bone-ash opals, which agrees with observations of Inwald,[16] who mentioned the advantage of using glasses with some lead content.

Another original method for enamels has been suggested to avoid roughness, excessive crystal growth and all of the difficulties derived from the high melting point of apatite crystals. Hermsdorf and Wagner[21] devised a method for enamels in which a mixture of two frits was used; one frit contained phosphoric acid in combination with alkali, alumina, and boric acid, and the other contained the divalent oxides, such as barium, calcium, or manganese, and some fluoride but no phosphorus pentoxide. Examples of these compositions are given in Table I.

Both batches were melted separately; when applied as a ground coat, 15 to 25 parts of frit No. 2 were added to 100 parts of frit No. 1 and mixed together. During the firing operation, the mixture melted to form a two-phase glass, and there was no apatite formation except at the interface. The relatively slow diffusion of the constituents prevented excessive crystallization.

TABLE I

	(1)	(2)
K_2O	9.2	5.3
Na_2O	12.2	14.3
Al_2O_3	10.0	5.9
P_2O_5	14.0	
B_2O_3	13.8	14.9
SiO_2	40.4	33.6
NiO	0.4	0.4
NaF		5.2
AlF_3		3.5
CaF_2		16.9

Efforts have been made recently to replace sodium borate by the sodium metaphosphate of domestic sources. Such a replacement in the average enamel would necessarily lead to the formation of apatite, inasmuch as enamels contain calcium and fluorine. According to Löffler,[22] the composition of the phosphate enamel has to be regulated so that it will not contain simultaneously appreciable amounts of alkaline earth and fluorine. As this particular enamel has been designed for antimony opacification, the melting point of any apatite phase would be lowered considerably.

[21] (*a*) L. Hermsdorf and R. Wagner, "Process of Enameling Ironware Using Calcium Phosphate to Increase Refractoriness and to Obtain Opacification," Ger. Pat. 166,672; Cl. 48*c*, July, 1904.
(*b*) L. Hermsdorf and R. Wagner, "Process of Producing an Enamel for Sheet-Iron Ware Using P_2O_5," Ger. Pat. 179,997; Cl. 48*c*, August 5, 1904.
(*c*) R. F. Wagner, "Enamels Which Contain Phosphates of Alkaline Earths," Ger. Pat. 179,440; Cl. 48*c*, February, 1905.

[22] J. Löffler, "Replacement of Borax by Na_5PO_4 in Enamels," Ger. Pat. 693,743; Cl. 48*c*, February 18, 1939.

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ELECTROLYTICAL REPLACEMENT OF SODIUM BY AMMONIUM IN GLASS*

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ABSTRACT

A review is given of the exchange of ions (1) in glasses containing water, (2) on the hydrated surfaces of ordinary glasses, and (3) in commercial glasses with and without the application of an electrical field, and it is shown that the ammonium ion offers unique features. It may be electrolytically introduced into soda glasses without impairing their structure as much as other ions. Because of the volatility of ammonium compounds, it may be removed easily, leaving a glass of higher silica content. Such glasses could be obtained in some experiments, but the soda probably cannot be entirely removed.

I. Introduction

(1) Base Exchange

The knowledge of the phenomena of ionic exchange in glass is drawn from two sources, namely, from the extensive study of base exchange proper and from work on electrolysis in glass. Even in its early stages, this work supplied information on the exchange of ions when glasses were in contact with amalgams, salt solutions, and salt melts. The extensive study of base exchange proper and the observations on surface reactions of glasses led to the conclusion that base exchange is a typical property of glass. In the field of crystals, however, only a few restricted groups like the zeolites show pronounced behavior of this kind.

The base exchange proper, that is, the exchange of ions without the application of an electric field, is usually governed by the laws of mass action.[1] Numer-

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[1] Ilse Zoch, "Base Exchange of Crystalline Zeolites with Neutral Salt Solutions," *Chem. Erde*, 1 [3] 219–69 (1919).

ous substances showing characteristic base exchange have been investigated by Schneiderhoehn,[2] who melted zeolitic minerals and, by quenching them, obtained glasses. These glasses have been found to show more rapid base exchange than the crystalline compounds from which they were derived. Unlike commercial glasses, they have a considerable water content and may be considered as intermediate between gels and commercial glasses. They have been used for water softening and for other purposes under the name of permutites.

Commercial glasses, too, may change their surface by reacting with water and by forming films which contain hydrated silicates. Similar exchange reactions, therefore, are to be expected on the surface of this type of glass. The surface of a commercial soda-lime glass, for example, is capable of adsorbing cations, such as K, Na, Ca, H, ammonium, and even organic bases like quinine.[3] These adsorbed cations may be reversibly replaced by calcium ions from solutions. In glasses which have been leached before the contact with the solution, these reactions may penetrate several molecular layers.

The formation of silver silicates on wet glass surfaces in contact with silver at room temperatures has been reported by different observers. The photochemical behavior of these silicates has led to the suggestion of "adsorption photography," which is a process[4] based on the fact that, by means of adsorbing silver ions on an ordinary glass plate, a layer sensitive to light may be developed without the use of gelatin or similar carriers.

The base exchange, however, is not restricted to the presence of hydrated layers; for example, at elevated temperatures, a direct exchange of ions may be observed. In contact with silver compounds and copper compounds and at temperatures not too far below the softening point, the glass exchanges sodium or potassium ions with silver or copper ions, which when reduced give rise to the well-known yellow silver stain and red copper stain. The reduction of silver is usually performed by the chemical effect of other compounds present in the glass; the reduction of copper requires the influence of reducing gases.

A remarkable base exchange between lithium and sodium ions has been observed whenever glasses are in contact with melts containing lithium ions. If, however, lithium ions replace sodium ions within the network of a silicate glass, they fill a much smaller place owing to their smaller size. These molecular holes easily cause supermolecular flaws and are thus responsible for the whitish appearance and the weakened structure of the glass which have been observed after such a contact. This property is so characteristic for lithium that a test for the presence of lithium has been suggested on this basis.[5]

Lithium compounds have been proposed as a substitute for hydrofluoric acid for etching glass.[6] For practical purposes, however, lithia etching has proved to be unsuitable because the base exchange causes fracture owing to the weakening of the structure. The decrease of strength observed in glasses decorated with glass enamels (for instance, bottles with enamel labels, the enamel containing lithia) is also caused by the same weakening process. This defect may therefore be observed even if the expansion coefficients of glass and enamel are perfectly matched. This is of particular importance because lithia has been used frequently as a constituent of chemically resistive glass enamels whenever a low alkali content with satisfactory fluxing properties is required. In such cases, intermediate layers are formed on firing whereby an exchange of lithium and sodium is favored.

(2) Electrolysis

Any exchange of ions in glass is greatly increased in amount and indeed is actually directed by the application of an electric field. Ever since the classical work of Warburg[7] and Tegetmaier[8] and the complete studies of Heydweiller and Kopfermann,[9(a)] the phenomena of ionic exchange in electrolysis have been well understood. The principal law governing the exchange of ions in the electrolysis of glass is that of Faraday, provided a sufficient exterior supply of ions is available. Its validity may be checked by inserting a silver voltameter in the electrical current. According to this law, the amount of ions accepted at the cathode and the amount of ions given up at the anode are equivalent to the silver deposited by the voltameter.

The possibility of such an exchange seems to depend on the similarity of charge and size of the ion in the glass and the ion supplied at the anode. When ions of different size are used, often no exchange or only a very small one is possible, and opacity and cracking of the glass result. The replacement of sodium by potassium therefore is difficult,[9(a)(b)] and the replacement of sodium by lithium leads to cracks[9(b)] as in the case of mere base exchange by contact which has been discussed. It is consistent with these considerations that potassium may be introduced easily into potassium glasses or lithium into lithium glasses.[9(b)]

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[2] H. Schneiderhoehn, "Base Exchange of Zeolitic Minerals," *Neues Jahrb. Mineral., Geol., Beilageband.*, **40**, 163–228 (1915).

[3] H. Devaux and E. Aubel, "Adsorption of Ions by Glass," *Compt. rend.*, **184**, 601–603 (1927); *Ceram. Abs.*, **7** [4] 259 (1928).

[4] F. L. Burmistrov, "Adsorption Photography," *Phot. Jour.*, pp. 452–59 (August, 1936).

[5] O. J. Stewart and D. W. Young, "Action of Molten Lithium Salts on Glass," *Jour. Amer. Chem. Soc.*, **57** [4] 695–98 (1935); *Ceram. Abs.*, **14** [10] 243 (1935).

[6] B. Siede, "Etching Effect of Lithium Nitrate on Glass," *Glas & Apparat.*, **18** [16] 163 (1937); *Ceram. Abs.*, **17** [5] 178 (1938).

[7] E. Warburg, "Electrolysis of Rigid Glass," *Ann. Physik*, **257**, 622 (1884).

[8] F. Tegetmaier, "Electrolytic Conduction of Glass and Rock Crystal," *Wied. Ann.*, **41**, 18 (1890).

[9] (*a*) K. Heydweiller and F. Kopfermann, "Glass Electrolysis," *Ann. Physik*, **32**, 739 (1910).

(*b*) C. B. Hurd, E. W. Engel, and A. A. Vernon, "Conduction Process in Glass: I, Replacement of Sodium by Alkali Metals and Ammonium," *Jour. Amer. Chem. Soc.*, **49**, 447–50 (1927); *Ceram. Abs.*, **6** [6] 218 (1927).

(*c*) C. A. Kraus and E. H. Darby, "Conduction Process in Ordinary Soda-Lime Glass," *Jour. Amer. Chem. Soc.*, **44**, 2783–97 (1922); *Ceram. Abs.*, **2** [5] 96 (1923).

Silver and copper ions may replace alkali ions with much higher speed when an electrical field is applied at high temperatures.[9]

II. Ammonium

Among the ions studied in electrolytical work have been Ba, Sr, Sn, Pb, Fe, and Co and, with negative results, Au, Pt, and uranyl.[9] The ammonium ion offers unique features. Ammonium compounds dissociate easily into gases when they are heated. The ammonium ion, in the opinion of the writers, was an ideal ion to be substituted into a finished glass and to be removed later in order to furnish a glass substantially poorer in alkali and richer in silica. Such a glass might also be shrunk at high temperatures in order to close the pores. A similar method of treating a porous intermediate product has already been used in the production of high silica glass.[10] A normally melting glass of special composition, in such a case, is manufactured, and after suitable shaping, it is changed in its structure by a heat treatment leading to the formation of two phases. One immiscible phase is soluble and may be leached out, leaving a skeleton of glass consisting of almost pure silica and still holding its original shape. This porous glass may be shrunk into a product of the same shape and its porosity may thus be removed.

Previous to the work of Hurd, Engel, and Vernon, [9(b)] practically nothing had been done on the replacement of sodium by ammonium in glass. They found that, among all of the volatile ammonium salts, saturated ammonium chloride solution and fused ammonium acetate were the most satisfactory for experiments at slightly elevated temperatures. An ordinary tungsten incandescent lamp was suspended in such a bath to provide the glass for the experiment. Such an arrangement was first described by Pirani and Lax[11] and used by Burt.[12] The glass of the incandescent bulb contained SiO_2 72.44, Na_2O 17.06, CaO 5.02, MgO 3.77, Al_2O_3 0.97, and Fe_2O_3 0.72% and also traces of arsenic. The filament of the incandescent lamp was the cathode, and a carbon rod the anode of the circuit, which was completed by the thermionic emission across the evacuated space of the lamp. This method has the advantage that the product liberated at the cathode is retained inside the bulb and may be easily examined.

Ammonia could be detected by the odor of the contents of the bulb when they were dissolved in water. The contents, furthermore, could be readily prepared for quantitative analysis. Pressure determinations supported the assumption, based on previous work of the writers, that the ammonium radical formed by the union of the emerging ion and an electron would dissociate into ammonia and hydrogen. The sodium that was present would form sodium amide and hydride.

At 110°C., the ammonium ions seemed to be able to traverse the 1-mm. thickness of glass without causing fracture. The glass was not discolored, but its surface cracked after several hours of continued electrolysis. The cracks showed a spider-web pattern, which increased in intensity when the glass was heated. When the heating was continued to dull redness, countless bubbles appeared along the cracks. With still longer time of electrolysis, fracture of the glass could be observed. The sodium ions could be replaced more readily by ammonium ions than by any of the other ions studied.

The earlier workers made no attempt to effect complete ionic replacement or to determine the degree of replacement. The present writers, however, intended to learn something about the possibilities of substituting the ammonium into a finished glass and to remove the ammonium by heat treatment in order to get a more siliceous glass. In judging these possibilities, it is necessary to know which percentage of the sodium can be replaced by ammonium and which conditions favor the optimum exchange while the glass still remains stable and does not suffer from an essentially weakened structure. Although the earlier investigators used temperatures between 100° and 200°C. because most of the ammonium compounds are volatile and unstable at high temperatures, the present writers decided that some experiments should be made at higher temperatures to determine the conditions and the maximum of ionic exchange for the particular purpose of obtaining a glass of high silica content.

III. Experiments of Electrolytic Replacement

The preceding considerations were helpful for choosing the salt to be used at the anode of the electrolytic apparatus. It was necessary therefore (*a*) that this salt melt somewhat below 200°C. and yet not dissociate below about 300°C. and (*b*) that it should contain ammonium as the only cation. In the case of ammonium salts, these specifications are rather limiting because of the tendency to dissociate. Salts with stable anions, like sulfates and phosphates, are the most suitable. The neutral ammonium sulfate, $(NH_4)_2SO_4$, starts to decompose into NH_3 and ammonium bisulfate, $(NH_4)HSO_4$, at low temperatures but the bisulfate is stable up to about 280°C. This temperature of stability was considered to be satisfactory for the present experiments. A mixture of this salt and concentrated sulfuric acid was chosen therefore because it melts readily and remains reasonably stable at the temperatures required; it also retains the ammonium ions after electrolytic treatment. In the choice of the anode salt, it is of primary importance that there be only the ammonium ion present at the temperatures used.

The Pirani and Lax method[11] of electrolysis offers some advantages for the experimental procedure, but its application is restricted to the use of lamp bulbs. The writers therefore preferred to be independent of the shape of the glass and to start with a pure sodium silicate glass. The glass used contained 78% of silica and was in the form of a test tube 12 in. long. This tube was filled almost to the top with a low melting

[10] H. P. Hood and M. E. Nordberg, "Production of High Silica Content Glass," Brit. Pat. 442,526, August, 8, 1934.

[11] M. Pirani and E. Lax, "Observations Made on Electrolytic Migration of Sodium Ions Through Glass," *Z. tech. Physik*, **3**, 232–35 (1922); *Ceram. Abs.*, **2** [4] 72 (1923).

[12] R. C. Burt, "Sodium by Electrolysis Through Glass," *Jour. Optical Soc. Amer.*, **11**, 87–91 (1925).

metallic alloy (Wood's metal). The specimen was placed inside a test tube of slightly larger diameter, and the intervening space was filled with the ammonium compound. The two tubes were placed in the furnace so that the specimen represented by the inner tube extended about 2 in. above the furnace. This was done in order to prevent the ammonium salt from "creeping" up the side and short-circuiting the system. A platinum wire was placed in the ammonium salt and a Nichrome wire in the Wood's metal. Direct voltages of between 6 and 115 could be applied but, after the preliminary experiments, interest was concentrated on the lower voltages which proved to be satisfactory for more complete replacements.

After the electrolysis had continued for about 2 hours, a white fine-meshed surface with a smooth milky appearance was noticed. As the time was extended and the electrolysis continued, this white area completely penetrated the wall of the sample tube, at which time the glass appeared perfectly white and opaque.

When samples of this white and opaque material were heated in an air-gas flame, a frothing and bubbling as of the evolution of gas was first noticed, after which the glass became rigid and more resistant to deformation. This was an indication that the resulting composition was nearer that of pure silica than was the original sodium-silicate glass.

The result of the analysis of a sample, which had been exposed to electrolysis for 4 hours at 280°C. and at 10 volts, showed 84.9% of SiO_2 and 12.5% of Na_2O, corresponding to a replacement of close to 45% of the Na_2O by ammonia. Different temperatures between 200° and 300°C. were tried and also different voltages, but 280°C. and 10 volts appeared to be the best. Not enough experiments have been made to decide which temperature and voltage will lead to the optimum value or whether complete replacement of the sodium is possible while the shape and strength of the glass is still maintained. These results prove, however, that at least one half of the sodium ions may be replaced. Under the conditions of these experiments, the structure of the glass has not been weakened enough to prohibit further treatment (e.g., shrinkage) or to prevent its use a a porous material, and it can be heated in a Bunsens burner without melting easily or cracking.

IV. Conclusions

These experiments demonstrate that it is possible to transform a glass into one of considerably higher silica content by exchanging some of its sodium ions and later by the removal of the ammonium and its derivatives. It is hoped that these results may stimulate further research on this subject.

For theoretical reasons, however, it is possible that all sodium ions do not behave in the same way under the influence of electrolysis and that perhaps not all of the sodium ions can be replaced without impairing the glass structure.

To explain the mechanical and electrical properties of glasses, Smekal[13] has introduced the conception that one portion of the sodium ions is bound by weaker forces than the others. By this conception, he interpreted the results of Tegetmaier[8] and of Kraus and Darby,[8(c)] who found the participation of sodium ions in electrolysis to be limited. The latter were unable to replace more than about 80% of the sodium ions by silver ions.

From the modern studies of the structure of glass by means of X rays, the electric field and the atomic configuration around each sodium may be expected to vary to a certain extent. It is possible, under the conditions examined in this experimental work, that only a part of the sodium ions can be removed whereas the rest would require much higher energies. The fraction of ions which can be easily replaced is a function of the composition of the glass, of the temperature, and of the previous history. Smekal shows that in the temperature region of the softening point about four fifths of all sodium ions have a rather high speed of migration whereas one fifth is difficult to remove.

Although the maximum amount of copper or silver ions introduced into the glass is not directly related to the problem of the completeness of Na-NH_4 exchange, it may be mentioned that Leibig and Hyde[14] were able to introduce 9% of copper or 13.3% of silver into glass by electrolysis. These amounts cannot be introduced into an ordinary silicate glass by batch addition and only certain phosphate glasses were found to dissolve as much as that. In acid aqueous solutions, the entire sodium content of a soda-lime-silicate glass could be exchanged with hydrogen ions by Haugaard.[15] An exchange of Ag from nitrate melts with the sodium of a glass takes place according to an equilibrium which is shifted to the side (silver in glass plus sodium in the nitrate melt). Repeated treatment of finely divided glass powder has been found to result in complete replacement of the sodium in the glass by silver from the melt.[16] In both cases, however, fine powders with a large surface have been used and the sodium bonds in the surface may not correspond to those in the interior of the glass.

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[13] Adolf Smekal, "Electric Conductivity and Constitution of Glass," *Glastech. Ber.*, **7** [7] 265–77 (1929); *Ceram. Abs.*, **9** [2] 89 (1930).

[14] E. C. Leibig and J. F. Hyde (Corning Glass Works), "Treating Glass Articles," U. S. Pat. 2,198,733, April 30, 1940; *Ceram. Abs.*, **19** [7] 163 (1940).

[15] G. Haugaard, "Action of Water on Glass," *Glastech. Ber.*, **17** [4] 104–106 (1939); *Ceram. Abs.*, **18** [8] 205 (1939).

[16] A. Guentherschulze and O. Mohr, "Distribution of Silver and Sodium Between Glass and Nitrate and Bromide Melts in Equilibrium," *Z. tech. Physik*, **13** [8] 356–58 (1932); *Ceram. Abs.*, **11** [12] 611 (1932).

AN INSTRUMENT FOR STUDYING FINE TEXTURE OF CERAMIC AND OTHER SURFACES*

By F. W. Adams, Lee Devol, and H. R. Letner

Abstract

The fine texture of various hard surfaces can be measured easily by using a tracing point and an optical lever in a profilograph of new design. Variations in the surface of 0.000001 in. may be detected and topographic or relief maps can be prepared at magnifications up to 6000×. The high accuracy is obtained with no more difficult machine work than the simultaneous lapping of two tapered bearings mounted on the same shaft. By supporting the tracing point and its mirror on torsion elements of special design, the instrument is rendered relatively insensitive to vibration and is given an unusually high resolving power. The kind of results obtained is illustrated by profilograms of chalkboards having varying writing and erasing characteristics and by a topography of a ground glass surface.

I. Introduction

It is often desirable to measure and study the microscopic contour of surfaces in which the texture has a depth of a few ten-thousandths inch or less from the tops of the hills to the bottoms of the valleys. Such measurements can be useful in studying (*a*) the surface produced in grinding plate glass preparatory to polishing, (*b*) etched surfaces used to diffuse light, and (*c*) surfaces of an abrasive character like those of the chalkboards now replacing the old-style blackboard in some schools.

The instrument described here[1] was designed primarily for use in studying the surface produced on plate glass by grinding, but it has proved useful in many other ways. When the need for such an instrument arose, a survey of the literature showed that there was no instrument available which would define the topography of a surface. The closest approach to this was a profilograph which had been constructed by Abbott[2] at the University of Michigan and a somewhat similar device which had been built in Germany by Schmaltz.[3] These instruments used a diamond stylus mechanically mounted on a mirror in such a way that movements of the diamond point in a direction normal to the surface of the material being studied could be magnified by an optical lever system and the results recorded on a photographic film. There were three serious deficiencies in these instruments, namely, (1) because of mechanical inaccuracies in the mechanism used to traverse the diamond across the surface, an elaborate scheme for compensation was necessary; (2) at high magnification, a limitation of resolving power seriously restricted the precision; and (3) these instruments were capable of tracing only a single profile and therefore were not adapted to define a topography.

II. Design of Instrument

An instrument was built to avoid the deficiencies of the earlier types which can be used to reproduce profiles at vertical magnifications of 1000 to 6000 and at horizontal magnifications of 50 to 700. Mechanical inaccuracies were avoided by a design in which only two simple mechanical motions are required for taking a profilogram. The resolving power was increased by providing several reflections from the mirror whose angle was controlled by the motion of the tracing point. Provision was included for tracing a series of horizontal profiles at definite intervals so that the exact topography of any area could be completely charted. A torsion element of simple design supported the mirror and tracing point so as to render the instrument relatively insensitive to vibration and at the same time to permit the use of a very light pressure of the tracing point against the surface.

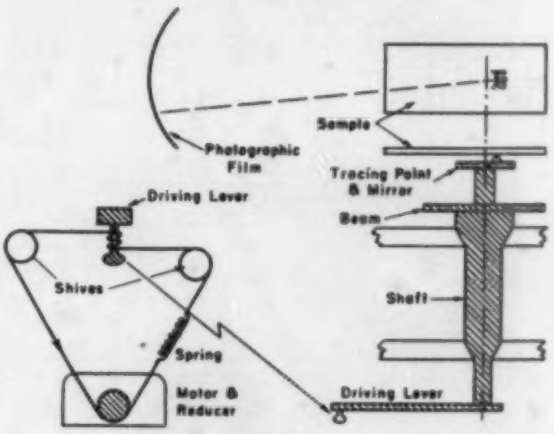

Fig. 1.

The instrument comprises three essential components, namely, (1) the shaft and driving mechanism, (2) the stylus and its suspension, and (3) the optical system. The general arrangement is shown in Fig. 1, which includes an elevation of the shaft-driving mechanism and a plan view of the sample and of the photographic film.

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* Presented at the Forty-Third Annual Meeting, The American Ceramic Society, Baltimore, Md., April 3, 1941 (Enamel Division). Received March 13, 1941.

[1] M. L. Devol, U. S. Pat. 2,205,517, June 25, 1940.

[2] E. J. Abbott and F. A. Firestone, *Mech. Eng.* (Sept., 1933).

[3] G. Schmaltz, Technical Surface Knowledge (Technische Oberflächenkunde), pp. 61 *et seq.* Julius Springer, Berlin, 1936; *Ceram. Abs.*, 16 [5] 141 (1937).

It was found that a bearing and shaft assembly could be constructed permitting the diamond stylus to be drawn accurately across the surface of the specimen through an arc of approximately 34 degrees. The shaft, $^3/_4$-in. in diameter, and bearings are mounted vertically, with about 5 in. between bearings. A horizontal beam is mounted on the upper end of the shaft to carry the optical system and the stylus suspension. This beam is arranged so that it can be moved horizontally to vary the radial distance of the stylus from the axis of the shaft and thus control the horizontal magnification of the instrument. Rotation of the shaft is accomplished by a small electric motor and speed reducer operating through a string drive to a lever arm attached to the vertical shaft.

III. Stylus Assembly

The stylus assembly is mounted on a suspension below the surface whose texture is being measured, the surface in turn being supported by four adjustable set screws, which permits it to be accurately leveled before making a trace. The stylus is a triangular pyramid having included angles between edges of approximately 60° and a point of 0.0002-in. radius. It is mounted on the back of a small front surface mirror which is suspended between two points on a yoke by torsion suspensions composed of 2-mil tungsten wire. By using two wires spaced approximately 0.1 in. apart at their connection to the yoke, it is possible to eliminate undesirable vibration and to provide sufficient rigidity to carry the weight of the mirror and stylus. The stylus is mounted approximately 0.07 in. off center from a vertical plane through the suspension points where the tungsten wires are fastened to the mirror. It is thus possible to reduce the force exerted by the stylus to less than 10 dynes and to provide the first lever in the optical system (see Figs. 2 and 3).

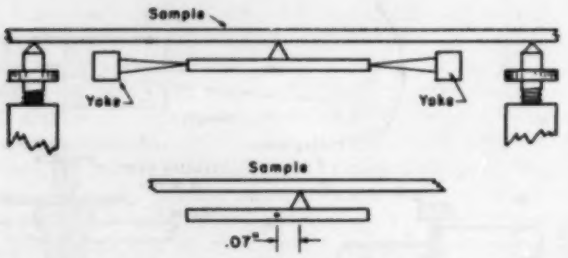

FIG. 2.

FIG. 3.—Adjusting stylus on topographic profilograph.

IV. The Optical System

Figure 4 shows the details of the optical system, where the moving parts are indicated by cross hatching. A concentrated filament lamp is focused on a pinhole approximately 0.001 in. in diameter. The light is then picked up by a condenser and sent through the optical system to focus on a photographic film. The light is carried by two prisms along the axis of the instrument shaft and thence by an auxiliary mirror to the stylus mirror. A front surface mirror fixed in the optical system and mounted below the stylus mirror picks up the light beam and reflects it back several times to the stylus mirror. By this multiple reflection from the mirror, a magnification of movement may be obtained without the long optical path that would otherwise be required and therefore with a smaller and brighter spot on the photographic film. After the final reflection from the stylus mirror, the light beam is picked up by another mirror which throws it off in a horizontal direction to a mirror mounted on the far end of the mechanical beam. From here, it is reflected back toward the axis of the shaft and then back again to a photographic film mounted on a radius of approximately 15 in. from the shaft. By the use of the complete optical system, a magnification of 6000 is readily obtained, with a line approximately 0.02 in. wide on the photographic film. By omitting the last two mirrors in the optical train, the magnification may be reduced two thirds (to approximately 2000). Further changes in magnification in the vertical direction are accomplished by adjusting the angle of the fixed mirror to alter the number of multiple reflections from the stylus mirror. Control of horizontal magnification is obtained by setting the diamond stylus at a suitable horizontal distance from the axis of the shaft.

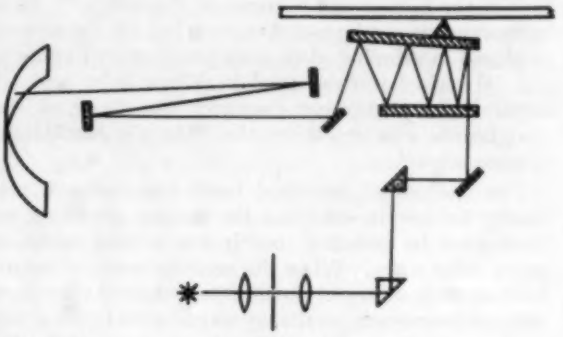

FIG. 4

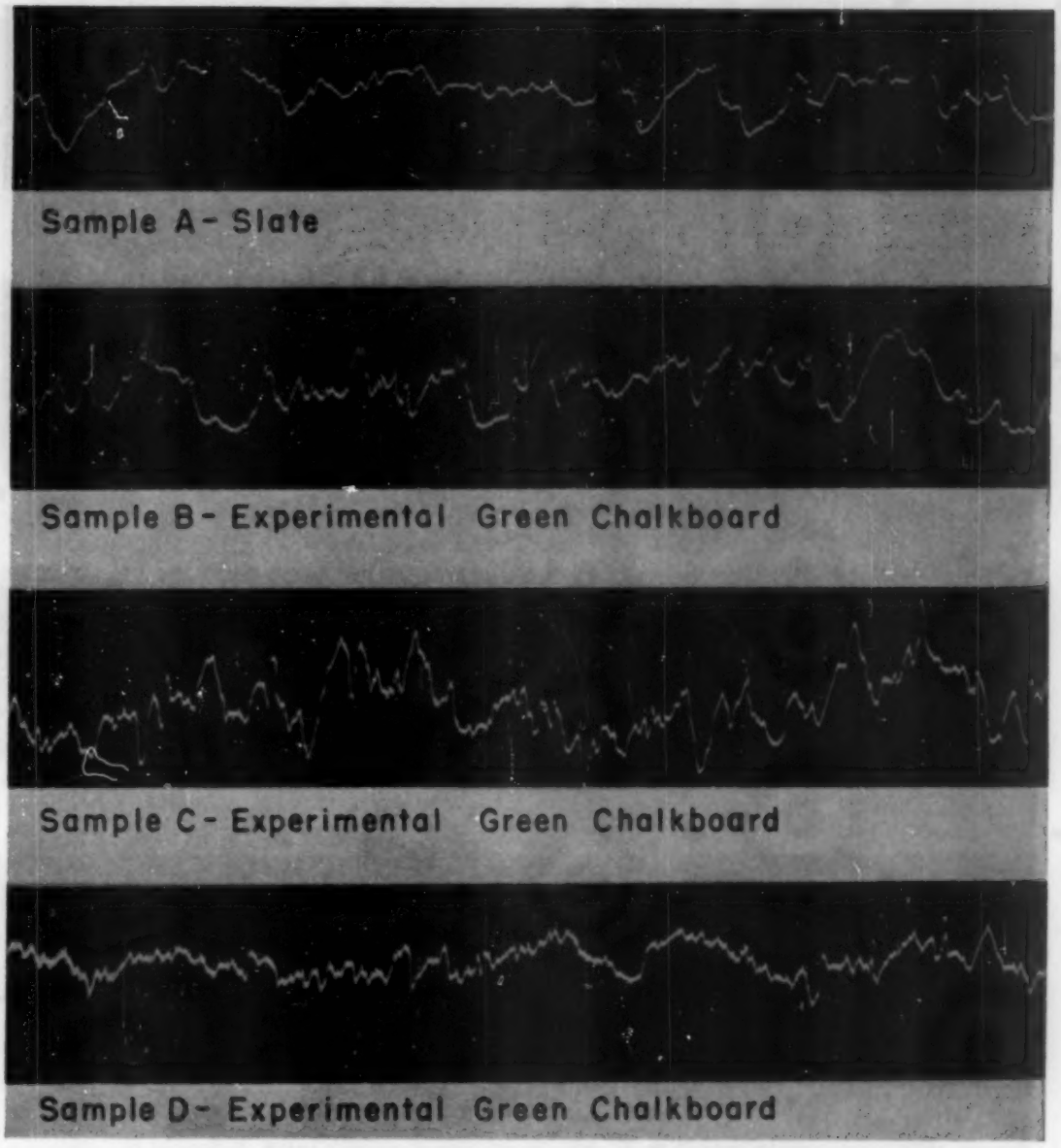
Sample A - Slate
Sample B - Experimental Green Chalkboard
Sample C - Experimental Green Chalkboard
Sample D - Experimental Green Chalkboard

FIG. 5.—Surface textures of chalkboards; magnification: vertical 1800×, horizontal 165×.

V. Use of the Instrument

The application of the profilograph to the study of surface textures is demonstrated by the development of a glass chalkboard of unique design. The desirable features of a surface for use in place of the common blackboard are (1) good writing characteristics, (2) easy erasure, (3) high diffuse reflectance, (4) absence of glare, and (5) permanence. Figure 5 shows profilograms at a vertical magnification of 1800 and a horizontal magnification of 165 of an ordinary slate blackboard surface and of three different experimental samples of green Nucite chalkboard. These chalkboards were made by flashing an abrasive grain in a vitreous binder on a plate-glass surface. On chalkboard sample B, the largest peaks and valleys have about the same size and character as those on slate (sample A). This chalkboard has good writing and erasure characteristics. It takes the chalk smoothly and erasure is easy. Sample C is rougher and has sharper peaks which are closer together. It is difficult to erase marks from this board, showing that this experiment points to an undesirable texture. Sample D, which is of a much finer texture, has good writing characteristics and an extremely mat surface.

A high diffuse reflectance is characteristic of light colors, provided the surface texture is designed to

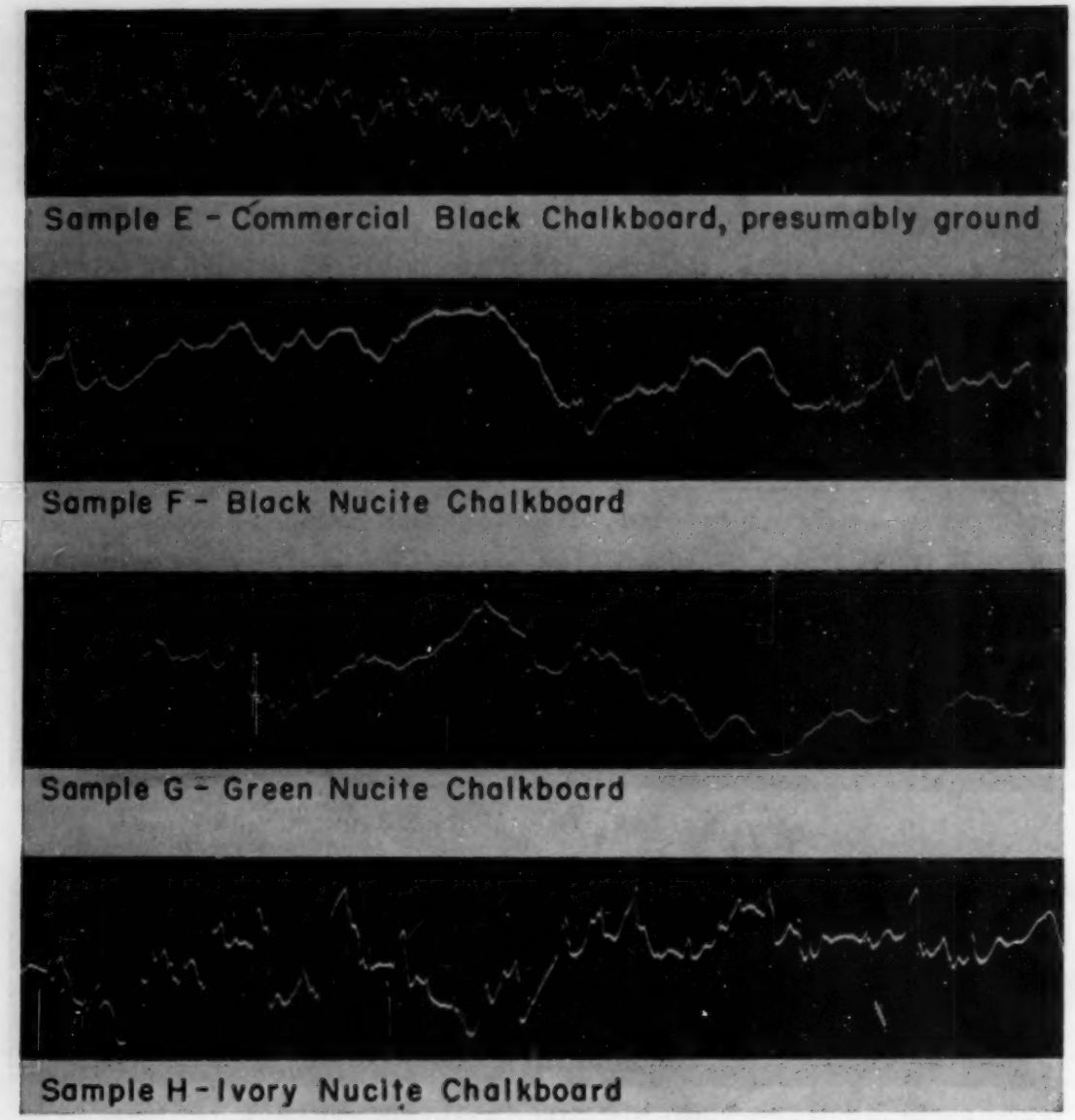
Sample E - Commercial Black Chalkboard, presumably ground
Sample F - Black Nucite Chalkboard
Sample G - Green Nucite Chalkboard
Sample H - Ivory Nucite Chalkboard

FIG 6 —Surface textures of chalkboards; magnification: vertical 1800×, horizontal 165×.

be free from glare. With the use of light-colored surfaces, therefore, freedom from glare is obtained by providing a thoroughly mat surface. This results in the reduction of eye-strain owing to a generous amount of diffuse illumination from a large area, and this may effect a substantial reduction in lighting costs. Figure 6 shows profilograms of four other chalkboard surfaces, which vary rather widely from one another in their profiles. The upper profilogram, sample E, is from a sample of black chalkboard of unknown construction picked up on the open market, but presumably with a finely ground glass surface. The others are of samples of commercial Nucite, prepared in the manner described previously. Figure 8 shows profilograms of the same surfaces after a 100-hour accelerated wear test, which is equivalent to about twenty years of normal service. Sample E shows that the peaks have been rounded off to produce a relatively smooth surface, which does not take chalk well, and it has a fairly high specular reflectivity, which produces an objectionable glare. A close examination of the other samples fails to reveal anything that is definitely indicative of wear.

To obtain a topographic picture of a surface, the profilograph is equipped to record a series of accurately spaced parallel traces. The diamond stylus can be moved known distances at right angles to the direction of a trace by shifting the position of the stylus assembly and optical system on the horizontal beam. This is

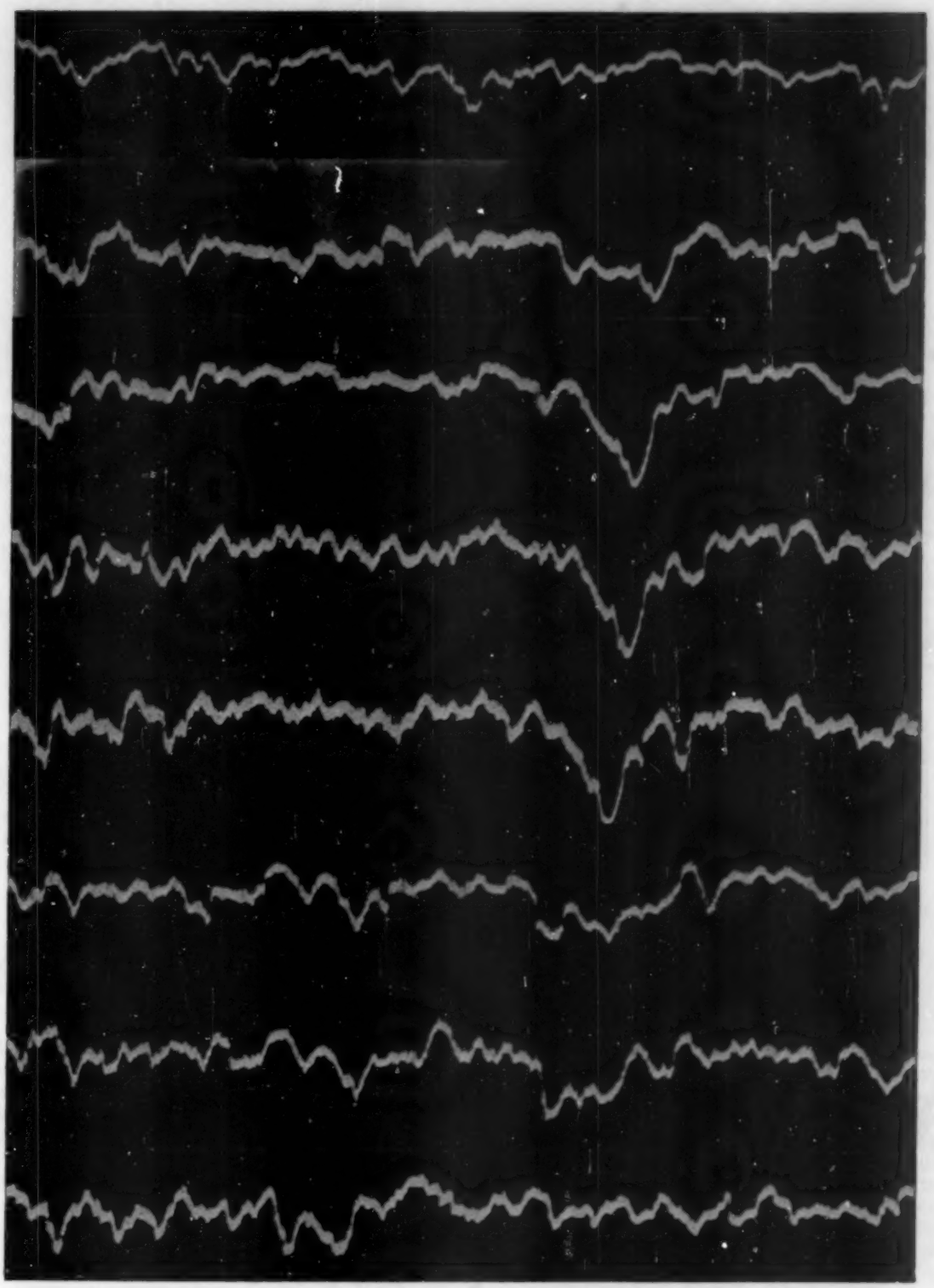

FIG. 7.—Topographic profilograms of a ground-glass surface; magnification: vertical 4800×, horizontal 400×; profile interval 0.00025 in.

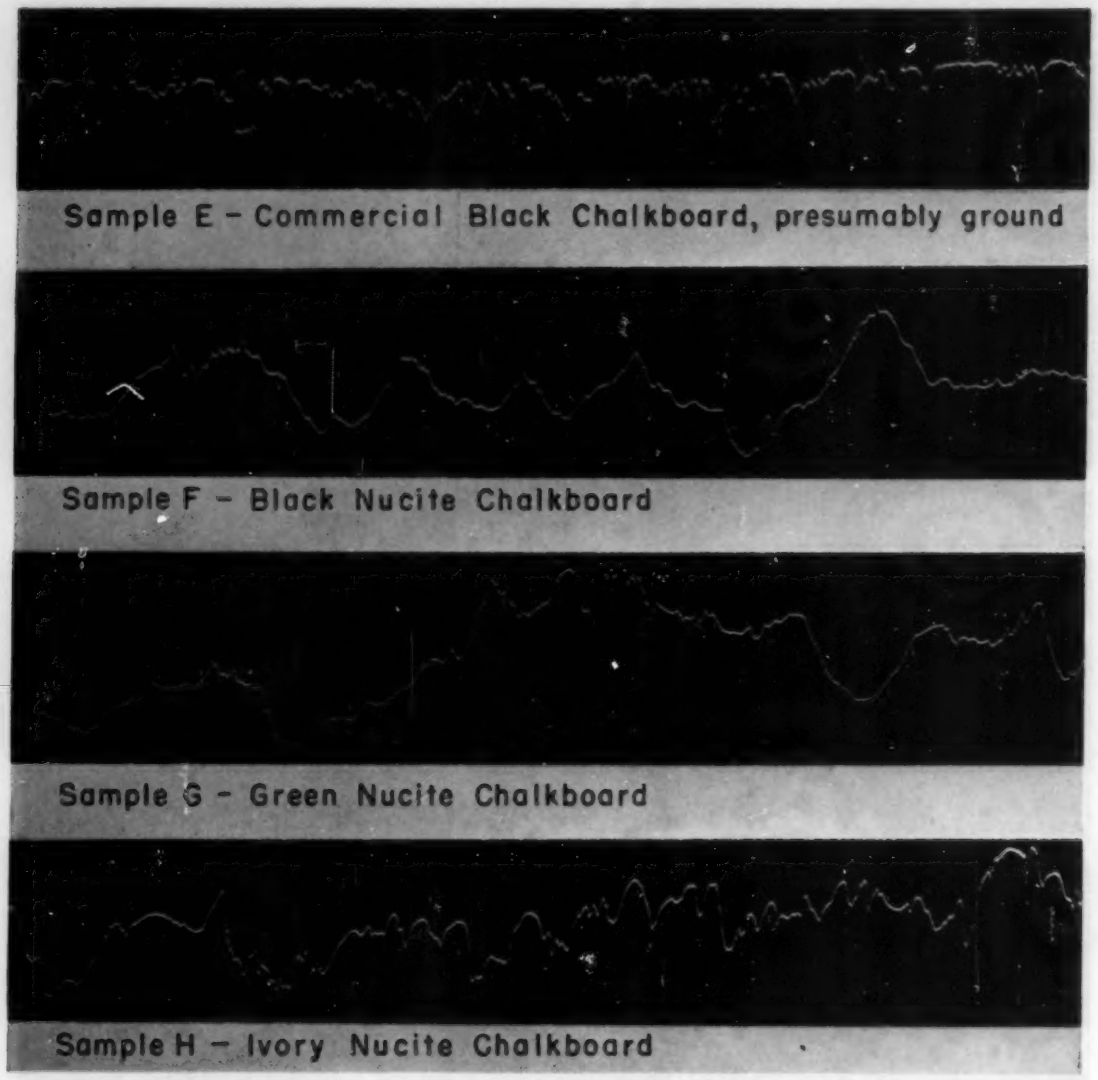
Sample E - Commercial Black Chalkboard, presumably ground
Sample F - Black Nucite Chalkboard
Sample G - Green Nucite Chalkboard
Sample H - Ivory Nucite Chalkboard

FIG. 8.—Surface textures of chalkboards after 100-hr. accelerated wear test; magnification: vertical 1800×, horizontal 165×.

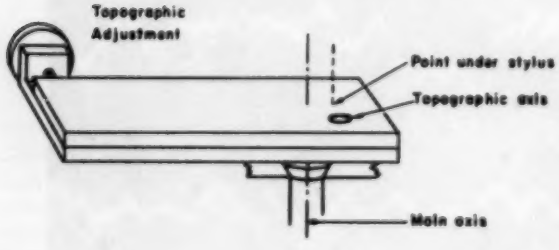

FIG. 9.

accomplished by an auxiliary pivoted beam and graduated micrometer screw (Fig. 9). A series of concentric traces, taken at definite small intervals, results in a series of profiles (Fig. 7), showing the topography of a sheet of ground glass at a vertical magnification of 4800, a horizontal magnification of 400, and a profile interval of 0.00025 in. It is obvious that a conventional topographic map or relief map may be constructed from these measurements.

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PRACTICAL THICKNESS OF PARTIAL COATINGS OF ACID-RESISTANT ENAMEL*

By George H. Spencer-Strong

Abstract

The effect of the firing cycle, fluidity of enamel glasses, water content of enamel slips, method of application, fineness of grind, and opacifiers on the actual thickness of partial acid-resistant enamel coatings, measured by the Shartsis and Harrison method, is discussed. The results of this study appear to explain the failure of highly acid-resistant enamels to produce satisfactory acid resistance in some enameling plants.

I. Introduction

Ever since the introduction of the partial or so-called "dust" coat of acid-resistant enamel, enamelers have been speculating as to just how much enamel is being applied. From 5 to 6 gm. per sq. ft. of enamel are known to give about 0.001 in. of enamel thickness, depending on the enamel and on the way it is handled. It has been argued, therefore, that a partial coating of 15 gm. per sq. ft. of acid-resistant enamel should give a coating of 0.0025 in. to 0.003 in. The partial coating, however, is applied over a wet coat of nonacid-resistant enamel so that the commingling of the enamels during the application and firing might be expected to be detrimental to the acid resistance.

Inasmuch as only surface tests for acid resistance have been used in the past, it has been impossible to determine the effect of the application of the partial coatings on the surface structure of the acid-resistant coating. Because many factors are known to affect the resistance of partial coatings of acid-resistant enamels, it is reasonable to surmise that some of these effects may be cumulative, and, although the individual factor of itself may be innocuous, a number of these apparently harmless factors combined may be extremely detrimental.

During the summer of 1939, it became necessary for Shartsis and Harrison of the National Bureau of Standards to determine the actual thickness of acid-resistant enamel coatings, and a simple and reasonably accurate method for such a determination was devised.[1] This method makes possible not only the actual measurement of the acid-resistant enamel but also the study of the interface between the acid-resistant and the nonacid-resistant enamels.

By using the Shartsis-Harrison test, studies may be made on various factors that affect partial coatings of acid-resistant enamels even though the factors of themselves are not sufficient to give rise to a visible effect on the acid-resistant properties of the surface. In the present investigation, the effect of six variable factors (commonly encountered in commercial practice) on partial coatings of acid-resistant enamel were studied as well as the effect of combinations of these factors on the enamel.

II. Plan of Investigation

The Shartsis-Harrison acid-resistant thickness test was used to determine the effect of firing time and temperature variations, variation of wetness of spray (for cover-coat and acid-resistant enamels), viscosity of enamels, fineness of milling, and specific gravity of enamel slips and opacifiers on the maximum and minimum penetration of acid-resistant coatings. Two enamels, a class A acid-resistant enamel of medium fusion flow and a superopaque nonacid-resistant enamel, were used for this study except for the effect of viscosity, in which case acid-resistant and non-acid-resistant enamels of comparatively short and long flows were used. Enamels were milled in accordance with the formulas shown in Table I; in the study of the effect of opacifiers, however, 6% of the opacifier in question was added to each enamel.

Table I

Mill Formulas of Enamels

Acid-resistant*		Nonacid-resistant*	
Frit	100 lb.	Frit	100 lb.
Clay	5 lb.	Clay	7 lb.
Ammonium carbonate	8 oz.	Magnesium carbonate	4 oz.

* Milled to 4% on 200-mesh.

To simplify the investigation, two application weights of acid-resistant enamel were chiefly used. Inasmuch as general practice in the industry stipulates a coating of 15 gm. per sq. ft. for partial acid-resistant coatings, the present study was carried on (1) at an application of 10 gm. per sq. ft., which is well below the minimum of good practice, and (2) at an application of 20 gm. per sq. ft., which is generally considered to be excessive. The acid-resistant coatings were applied over a wet partial coating of a nonacid-resistant cover-coat enamel, which in turn had been applied over a fired coating of 32½ gm. per sq. ft. of a nonacid-resistant cover-coat enamel. The total application weight in each case was 65 gm. per sq. ft., and the application of the wet nonacid-resistant coating was varied with the application weight of the acid-resistant enamel so that the total second coat in each case was the equivalent of a

* Presented at the Forty-Second Annual Meeting, The American Ceramic Society, Toronto, Canada, April 10, 1940 (Enamel Division). Received July 22, 1940; revised paper received January 21, 1941.

[1] W. N. Harrison and Leo Shartsis, "Determination of Thickness of Acid-Resistant Portion of Vitreous Enamel Coatings," *Jour. Research Nat. Bur. Stand.*, **25** [1] 71–74 (July, 1940); R. P. 1315; *Ceram. Abs.*, **19** [11] 253 (1940).

coating of 32½ gm. per sq. ft. For example, when a coating of 10 gm. per sq. ft. of acid-resistant enamel was desired, it was necessary to apply the nonacid-resistant cover-coat at 22½ gm. per sq. ft. Enamels were applied over 4- by 6-in., 18-gauge enameling iron plaques covered with 2½ oz. per sq. ft. of commercial ground coat. Nonacid-resistant cover-coat enamels were fired for 2½ minutes at 1500°F., and the acid-resistant coating was fired for 2½ minutes at 1525°F. During the investigation of the effects of firing time and temperature on the acid-resistant coating, however, the firing treatment was as noted.

At the conclusion of the experiments, which were designed to show the effect of various individual factors on the thickness of the acid-resistant coating, the composite effect of the various factors was investigated. Two sets of sample plates were made up, the first to have a maximum amount of acid-resistant enamel thickness with a minimum enamel coating and the second to have a minimum actual thickness of acid-resistant enamel with a maximum application. In both series, the acid-resistant enamels were applied at weights ranging from 8 to 16 gm. per sq. ft. in increments of 2 gm. per sq. ft. with the weight of the nonacid-resistant coatings varied to maintain a total cover-coat enamel coating of 65 gm. per sq. ft. The resulting plaques were tested for acid resistance in accordance with the Standard Method of the Porcelain Enamel Institute (as revised in April, 1940) to determine the application weight of acid-resistant enamel required to secure a class B, or better, acid resistance with each series.

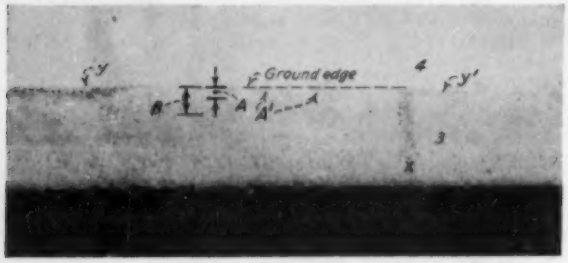

FIG. 1.—Comparison of methods of marking ground edge.

III. Thickness Test Method

Enameled panels were cut into 2-in. squares. The enamel along one sheared face was ground on a cast-iron lap (using 200-mesh Alundum mixed with water to form a thin paste) at an angle of approximately 5 degrees to the face of the sample to expose an area approximately 10 mm. wide. After grinding, the total width of the exposed enamel, including the ground coat, was measured with a low-power microscope to the closest 0.1 mm., and the total enamel thickness was measured with the General Electric thickness gauge to ±0.0005 in. The ground edges of the specimen were marked to show the original edge of the ground surface, and the specimen was refired just enough to give a fire polish to the ground areas. Samples were immersed in a fresh solution of 10% citric acid for 15 minutes. During this treatment, the acid attacked the exposed nonacid-resistant enamel but not the acid-resistant enamel, so that when a soft lead pencil or china marking crayon was rubbed across the treated area, the resulting marks were retained on the etched, nonacid-resistant portion of the beveled surface but were readily wiped away from the acid-resistant surfaces. The width of the exposed acid-resistant enamel layer was then measured, and the actual thickness of the acid-resistant coating was determined from the following formula:

$$h_A = h_T \times \frac{W_A}{W_T}.$$

Where h_A = thickness of acid-resistant coating.
h_T = total thickness of enamel.
W_A = width of exposed acid-resistant enamel.
W_T = total exposed enamel width.

By the Shartsis-Harrison method, the edge of the ground area should be marked by running a ceramic pencil along the edge of the cut before fire polishing. The material of the pencil adheres to the ground area but not to the surface of the specimen. After the fire-polishing operation, the resultant line is fired into the enamel, making a permanent marking.

During the progress of the investigation, it became apparent that the presence of this marking line interfered with the measurement of the minimum acid-resistant thickness, especially when the thickness was small, and it was necessary to change the method of marking. To accomplish this, several parallel lines were drawn with a ceramic pencil across the ground area from the exposed metal to the original surface. The lines readily showed the edge of the ground area, inasmuch as the pencil marks adhered to the ground surface but not to the glossy surface.

Figure 1 shows a comparison between the original method of marking the entire edge with a china marking pencil and the improved method described. The horizontal lines, y, at the extreme left, and, y^1, right, obtained by using the original method, show the type of results that may be expected. Areas marked 1, 2, 3, and 4 are the iron, ground-coat, nonacid-resistant cover coat, and acid-resistant coat, respectively. The maximum acid-resistant enamel thickness is shown at B, and A shows the minimum acid-resistant enamel thickness. Lines y and y^1 were purposely carried only part way across the specimen to show their effect on the measurement of the coating thickness. Even where the coating is relatively thick, as at A, the true thickness would be obscured by y; y^1 (which represents the thinnest line possible to produce on this type of surface) obscures very thin acid-resistant coatings, for example, A^1. The vertical line, X, representing the improved method, does not interfere with the measurement of the acid-resistant coatings.

The range of the results which may be expected by using the Shartsis-Harrison method to measure the thickness of acid-resistant coatings is shown in Figs. 2, 3, and 4. For purposes of comparison, the acid-resistant enamel coatings were applied at 10 gm. per sq. ft. A dust coating of poor acid-resistant quality

is shown in Fig. 2. In Fig. 3, a similar dust coating is shown, prepared from the same enamels but with a different application technique in which excellent protection is obtained. Note the relatively great penetration of the acid-resistant enamel into the nonacid-resistant base coat in Fig. 2 as compared to Fig. 3.

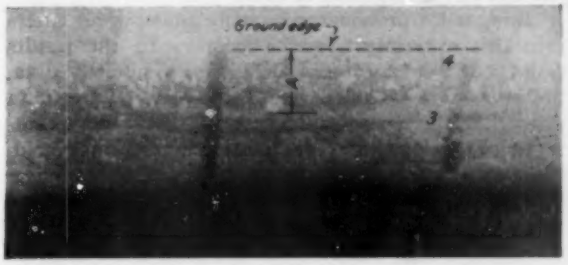

FIG. 2.—Acid-resistant enamel application of 10 gm. per sq. ft. over wet nonacid-resistant cover coat shows extreme intermingling between acid-resistant and nonacid-resistant enamels; (1) base metal, (2) ground coat, (3) nonacid-resistant cover coat, and (4) acid-resistant enamel; *A*, maximum depth of penetration of acid-resistant enamel; note nonacid-resistant enamel extending to surface of acid-resistant coating, indicated by dark areas above the ground edge of the panel.

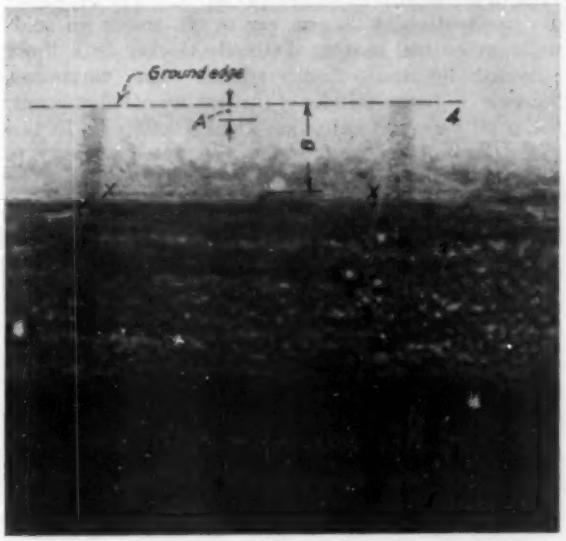

FIG. 3.—Application of 10 gm. per sq. ft. of acid-resistant enamel over wet cover coat; same specimen enamels as those used in Fig. 2; minimum thickness of acid-resistant coating at *A* and the maximum penetration at *B*; note improvement in acid-resistant coating over that shown in Fig. 2.

This dispersion naturally reduces the density of the surface acid-resistant coating; in some cases, as in Fig. 2, it penetrates so deeply as to allow the nonacid-resistant enamel to extend to the surface of the piece and thus destroys its acid-resistant quality. The technique used in the preparation of specimen 3 reduces this penetration, giving a denser coating of acid-resistant enamel and therefore adequate protection. Figure 4 shows the effect of the application of a coating of acid-resistant enamel over a fired base coat as compared with the wet base coat shown in Figs. 1, 2, and 3. Note that the commingling of the two coatings is restricted to the natural irregularities on the surface of the original coating so that, in comparing the enamels applied by the two methods, a practically smooth surface is obtained when the acid-resistant enamel is applied over a fired base coat, whereas a ragged surface interface is obtained with the application over the wet base.

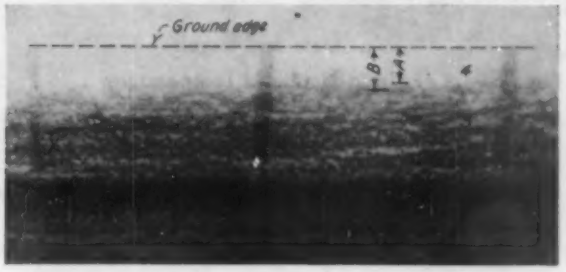

FIG. 4.—Effect of applying acid-resistant enamel over a fired coating; minimum acid-resistant coating thickness *A* and the maximum penetration acid-resistant enamel *B* are almost identical; note the relatively smooth interface between acid-resistant enamel 4 and nonacid-resistant enamel 3 as compared with the rough interface shown in Figs. 2 and 3; the maximum penetration of the acid-resistant enamel *B* is much less than the similar penetration shown in Figs. 2 and 3.

(1) Accuracy of Test

The accuracy of the Shartsis-Harrison test depends chiefly on three factors, namely, (1) the depth of the ground area, (2) the accuracy of measurement, and (3) the physical nature of the enamel. The General Electric thickness gauge in the range used in this test (that is, 0.015 in. total enamel thickness) has an accuracy no greater than ±0.0005 in., depending on the care used by the observer. With a 10-mm. ground area, a maximum variation of 0.000125 in. is encountered per 0.001 in. of error in the reading of the thickness gauge. The error increases with the increased thickness of the acid-resistant enamel, and with a 5-mm. area, the error is doubled, which would indicate that the wider the ground area the less the effect of errors in thickness measurement. If, however, the ground area is too wide, the interface between the acid-resistant and the nonacid-resistant enamels is distorted to such an extent that accurate measurement is extremely difficult. Errors in the measurement of the width of the ground area may also cause serious errors in the results. Thus, an error of 0.1 mm., at 15 mils of enamel thickness and with a ground area 10 mm. wide, will cause an error of about 0.00015 in. As the length of the ground area decreases, the errors resulting from inaccurate measurements increase. Under certain conditions, the interface between the acid-resistant and the nonacid-resistant enamel may be extremely rough, presenting a cerrated effect, and sometimes the layer may be vesicular with bubbles or globules of nonacid-resistant enamel penetrating into a seemingly solid acid-resistant coating. Thus accuracy in judgment as

to just what is the true thickness of the acid-resistant coating is necessary. Although Shartsis and Harrison have indicated that the method is accurate approximately to 0.0005 in., in most cases the accuracy may be even greater. For the present investigation, however, variations in thickness of more than 0.0005 in. between individual data were considered to be significant; variations of 0.00025 to 0.0005 in. were taken to be indicative of trends, and variations of less than 0.00025 were considered to be within the error of the method and therefore inconsequential.

IV. Data and Results

(1) Effect of Firing Time and Temperature

Specimens were fired for $2^1/_2$ and 5 minutes at 1500°, 1525°, and 1550°F., respectively (Table II). The variation in firing time showed little effect except at 1550°F., where significant gains were noted both in maximum and minimum thickness of the acid-resistant enamel. At 1500°F., the increased firing time

TABLE II

EFFECT OF FIRING TIME AND TEMPERATURE ON MAXIMUM AND MINIMUM THICKNESS

Firing temp. (°F.)	Acid-resistant coating thickness (mils) applied at 10 gm./sq. ft. (Max.)	10 gm./sq. ft. (Min.)	20 gm./sq. ft. (Max.)	20 gm./sq. ft. (Min.)
	A ($2^1/_2$-min. cycle)			
1500	2.53	1.09	4.14	2.86
1525	3.25	1.19	4.35	3.20
1550	3.33	1.32	5.42	3.88
	B (5-min. cycle)			
1500	2.89	0	4.00	2.74
1525	3.04	1.53	4.54	2.85
1550	4.21	1.98	5.90	4.00

appeared actually to decrease the minimum thickness of the acid-resistant coatings, and at higher temperatures, the coating was increased by the longer firing time. The increase in the firing temperature gave rise to a slight increase in the enamel thickness at $2^1/_2$ minutes of firing and to a significant increase in the acid-resistant coating thickness at 5 minutes.

(2) Effect of Spraying Method

The wetness of the spray of the acid-resistant and nonacid-resistant enamels was varied by changing the distance from the nozzle to the plaque as well as by the atomization of the gun. Attempts were made to determine the effect of wet, medium, and dry spray of both enamels in various combinations.

Owing to the pronounced human element which enters into this experiment, great accuracy in the results could not be expected. Definitely significant data, however, were obtained (see Table III). These data show that a maximum thickness of acid-resistant coating is obtained with a combination of dry-spray acid-resistant and dry-spray base coatings. In applications of 10 gm. per sq. ft., only the dry-over-dry and the dry-over-medium show a completely acid-resistant enamel coating. Acid-resistance tests show that the medium-over-dry and the medium-over-medium applications give the best protection to the remainder of the specimens. The use of a wet acid-resistant coating over all three types of nonacid-resistant coatings give the poorest results, the worst of the lot being a wet acid-resistant enamel over wet nonacid-resistant enamel. In this case, as in a number of other instances in this investigation, the effect of the various types of coatings applied at 20 gm. per sq. ft. is not so marked as in those applied at 10 gm. per sq. ft. The dry-over-dry application at 20 gm. per sq. ft. shows an acid-resistant enamel coating distinctly thicker than those obtained by the other methods. The variations, however, in the remainder of the test are not significant, the total variation being less than 0.0005 in. In this case, the wet-over-wet application again appears to show minimum acid-resistant enamel thickness.

(3) Fineness

Acid-resistant and nonacid-resistant enamels, milled to 1% of fineness on 200-mesh and to 6% on 200-mesh, were applied in various combinations (Table IV). All of the coatings of 10 gm. per sq. ft. show complete acid-resistance, and the variation between the coatings is not significant. There is, however, a definite trend which indicates that the fineness of the base coating

TABLE III

EFFECT OF SPRAYING VARIATIONS ON MAXIMUM AND MINIMUM THICKNESS

Spray dryness (1)*	Spray dryness (2)†	Acid-resistant coating thickness (mils) applied at 10 gm./sq. ft. (Max.)	10 gm./sq. ft. (Min.)	P.E.I. acid-resistance rating‡	20 gm./sq. ft. (Max.)	20 gm./sq. ft. (Min.)
Dry	Dry	2.75	1.55	A (1)	4.32	2.88
"	Medium	2.96	1.03	A (2)	4.13	2.52
"	Wet	2.75	0	AD (5)	3.65	2.18
Medium	Dry	2.51	0	" (3)	4.29	2.45
"	Medium	2.58	0	" (4)	3.60	2.44
"	Wet	2.65	0	" (6)	5.09	2.51
Wet	Dry	2.59	0	" (7)	4.10	2.16
"	Medium	3.17	0	" (8)	4.40	2.53
"	Wet	3.79	0	" (9)	5.01	2.10

* Acid-resistant coat.

† Nonacid-resistant coat.

‡ Double letters, AD, denote small areas with class D acid resistance, whereas the major portion shows class A acid resistance; numerals denote comparative acid resistance between specimens.

has more effect on the acid-resistant coating thickness than a variation in the fineness of the cover coat. With the application of 20 gm. per sq. ft., only the combination of the coarsely milled enamels shows a significant increase in thickness of coating.

TABLE IV

EFFECT OF ENAMEL FINENESS ON MAXIMUM AND MINIMUM THICKNESS

Residue on 200-mesh sieve (gm.)		Acid-resistant coating thickness (mils) applied at			
		10 gm./sq. ft.		20 gm./sq. ft.	
(1)*	(2)†	(Max.)	(Min.)	(Max.)	(Min.)
6	6	2.61	1.24	4.60	3.22
1	6	2.77	1.24	4.20	2.52
1	1	2.35	0.83	3.54	2.18
6	1	2.36	0.73	4.07	2.62

* Acid-resistant coat.
† Nonacid-resistant coat.

(4) Viscosity

The effect of the comparative molten viscosities of enamels on the thickness of the acid-resistant coating, determined by the fusion flow method, was investigated. Base enamels with fusion flows of 40 and 64 mm. were applied in various combinations with acid-resistant enamels having fusion flows of 35 and 41 mm., respectively (see Table V). The data indicate that with an application of 10 gm. per sq. ft. only the

TABLE V

EFFECT OF ENAMEL VISCOSITY ON MAXIMUM AND MINIMUM THICKNESS

Fusion flow (mm.)		Acid-resistant coating thickness (mils) applied at			
		10 gm./sq. ft.		20 gm./sq. ft.	
(1)*	(2)†	(Max.)	(Min.)	(Max.)	(Min.)
41	64	2.71	1.00	4.25	3.23
35	40	2.78	0	4.22	2.29
35	64	2.83	0	3.55	1.45
41	40	2.96	0	4.41	3.32

* Acid-resistant coat.
† Nonacid-resistant coat.

combination of enamels of low viscosity shows a complete coating. For applications of 20 gm. per sq. ft., the viscosity of the acid-resistant enamel appears to be the governing factor because when acid-resistant enamels of long flow were used the maximum acid-resistant enamel thickness was obtained and the coatings were of similar thickness in both cases. When acid-resistant enamels of high viscosity were used, the total thickness of the acid-resistant coating was markedly thinner, especially when the high-viscosity acid-resistant enamel was combined with the low-viscosity nonacid-resistant enamel.

(5) Specific Gravity

Acid-resistant enamels set up to specific gravities of 1.85 and 1.72 and nonacid-resistant enamels set to specific gravities of 1.80 and 1.68 were applied in various combinations (Table VI). The data indicate that in the application of 10 gm. per sq. ft. only the combination of the enamels sprayed at high specific gravity gives a completely acid-resistant coating which is none too thick. The combination of the low specific gravity acid-resistant enamel with the high specific gravity base coating gives the second-best results, as determined by the comparative acid resistance of the enamels. With a coating of 20 gm. per sq. ft., the variations again are not nearly so significant as in the coatings of 10 gm. per sq. ft. The high specific gravity base coating, moreover, gives the best results in combination with the high specific gravity acid-resistant enamel; the low specific gravity base coating gives the poorest result.

TABLE VI

EFFECT OF SPECIFIC GRAVITY OF MILLED ENAMELS ON MAXIMUM AND MINIMUM THICKNESS

Sp. gr.		Acid-resistant coating thickness (mils) applied at				
		10 gm./sq. ft.		P.E.I. acid-resistance	20 gm./sq. ft.	
(1)*	(2)†	(Max.)	(Min.)	rating‡	(Max.)	(Min.)
1.846	1.800	3.22	0.67	A (1)	3.97	2.56
1.846	1.681	3.00	0	AD (3)	4.34	1.87
1.723	1.800	2.80	0	AD (2)	4.03	2.45
1.723	1.681	2.96	0	AD (4)	4.55	2.44

* Acid-resistant coat.
† Nonacid-resistant coat.
‡ Double designation, AD, denotes presence of small areas with an acid resistance of class D; the major portion of the surface showed a rating of class A; numerals indicate the relative acid resistance of the specimens.

(6) Opacifiers

A comparison was made between enamels milled (*a*) with no opacifier and (*b*) with three types of commercial opacifiers, 6% of opacifier being used in each case. No effort was made to determine the effect of the various combinations of opacifiers, for example, a coating of opacifier A over opacifier B (Table VII). There is little or no significance to the results except that opacifier C, in the coatings at 10 gm. per sq. ft. and 20 gm. per sq. ft., shows a decreased acid-resistant coating thickness and opacifiers A and B appear to increase slightly the acid-resistant enamel coating.

TABLE VII

EFFECT OF OPACIFIER ON MINIMUM AND MAXIMUM THICKNESS

Opacifier	Acid-resistant coating thickness (mils) applied at			
	10 gm./sq. ft.		20 gm./sq. ft.	
	(Max.)	(Min.)	(Max.)	(Min.)
None	3.25	1.16	4.36	3.20
A	3.41	1.40	4.78	3.44
B	3.17	1.41	4.62	3.22
C	2.80	1.05	4.04	2.61

(7) Effect of Combinations of Factors Studied on Coating Thickness

To investigate the effect of combinations of factors on the acid-resistant coating, two sets of enamels were prepared as follows: (*a*) a combination of those factors which should give a minimum thickness of actual acid-resistant enamel coating with a maximum applica-

tion weight and (*b*) a combination of factors which should give a maximum thickness of acid-resistant enamel coating with a minimum application weight. In other words, with the first combination of properties in (*a*), the acid-resistant enamel should reach a minimum efficiency, whereas with the combination in (*b*) maximum efficiency is obtained. (*a*) The data indicate that an acid-resistant enamel of low molten viscosity milled at 6% on 200-mesh and sprayed wet at a high specific gravity over a nonacid-resistant cover-coat enamel and also one having a low molten viscosity milled at 1% on 200-mesh and sprayed wet at a low specific gravity should produce a minimum thickness of acid-resistant enamel at a maximum application weight; and (*b*) an acid-resistant enamel of low molten viscosity milled to 6% on 200-mesh and sprayed dry at a high specific gravity over a nonacid-resistant cover-coat enamel of medium molten viscosity milled to 6% on 200-mesh and sprayed dry at a high specific gravity should give a maximum thickness of acid-resistant coating at a minimum application weight.

The results of these tests show that a coating of 16 gm. per sq. ft. of combination (*a*) is required to give a class B acid-resistant finish, whereas a coating of 8 gm. per sq. ft. of combination (*b*) gives a class A enamel. This experiment significantly demonstrates the pronounced effect of the combination of factors affecting acid-resistant enamels on obtainable results.

V. Discussion of Results

The results of the investigation indicate that a combination of the various factors studied produces far more significant changes in acid-resistant coating thickness than any single factor. The dryness of spray and, as correlative factors, the spray pressures, firing treatment, specific gravity of slips, molten viscosity of enamels, fineness of grinding, and opacifier content affect the acid-resistant coating thickness in the order named. The application method and firing temperature appear to be the most important of these factors. The behavior of extremely thin coatings (10 gm. per sq. ft.) differs widely from that of heavier coatings (20 gm. per sq. ft.). The minimum thickness of acid-resistant enamel which actually gave a complete coating was about 0.00075 in. With these coatings, bubbles from the nonacid-resistant portion of the coat tend to disrupt the continuity of the acid-resistant enamel and to cause specks under the acid-resistance test; that is, a class D acid resistance is noted in the area where the bubbles burst, whereas a class A coating is obtained over the remainder of the piece. The ratio of total penetration of an acid-resistant enamel to the minimum coating thickness is much greater in an application of 10 gm. per sq. ft. than at 20 gm. per sq. ft. and, as a result, the 10-gm. applications were much more susceptible to the variations of the test. Because there is a marked penetration of the acid-resistant enamel into the nonacid-resistant base coating with partial acid-resistant enamel coatings that is not encountered with full coatings, the same thickness of a solid acid-resistant coating, on the basis of the weight of the acid-resistant enamel actually applied, cannot be produced. Conversely, even though the solid acid-resistant coat that results from the application of partial coatings may not be so thick as that from the application of a full coat of acid-resistant enamel over a fired nonacid-resistant enamel owing to the commingling of the enamels, the effective thickness of the acid-resistant coating is considerably increased. In other words, although the acid-resistant coating may not be a solid class A coating, the penetration of the acid-resistant enamel into the underlying cover coat produces in effect a full coating of semiacid-resistant enamel, which will give a great deal more protection than the normal cover coat. When the surface has been destroyed because of abrasion and other factors, the enamel product normally becomes unsightly and is discarded; the acid resistance of the surface is naturally a factor of paramount importance. There are some applications, however, wherein the appearance factor is not important; the penetration of the acid-resistant enamel into the nonacid-resistant enamel is important in these cases.

VI. Conclusions

(1) The actual thickness of solid acid-resistant enamel coatings, produced by the partial-coating method, is not equivalent to the thickness of the coating produced by the same application over a fired nonacid-resistant enamel.

(2) The intermingling of the acid-resistant and of the nonacid-resistant enamels during application by the partial-coating method produces a semiacid-resistant coating which is practically the equivalent of the combination of the acid-resistant and nonacid-resistant coatings.

(3) A combination of factors encountered in the production of partial acid-resistant coatings produces far more significant changes in the acid-resistant coating than any single factor.

(4) The application method and firing temperature have the greatest individual effect on the thickness of acid-resistant enamel coatings applied by the partial-coating method.

(5) The specific gravity of slips, the molten viscosity of the enamels, the fineness of grinding of the acid-resistant and nonacid-resistant cover coats, and the opacifier contents affect the acid-resistant coating thickness in the order named.

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FACTORS INFLUENCING DRY-BEADING DEFECTS*

By R. J. Whitesell

Abstract

The effect of milling and of application variables on the dry-beading defects of two types of kitchenware enamels was determined. The tearing adjacent to the bead was influenced by the same factors that govern the usual tearing encountered in all enamels. The extent of blistering was controlled by the quantity and nature of the soluble salts deposited in the water line. Potassium carbonate was a primary cause of blistering. Under certain conditions, however, the addition of sodium nitrite or sodium thiosulfate either to the cover enamel or to the beading enamel eliminated blistering.

I. Introduction

One cover-coat kitchenware is usually dry beaded. The beading enamel is applied to the beads and handles after the cover coat has been dried. There are two defects common to this method of beading. The first is a depressed, usually blistered, band, which marks the deepest penetration of the beading enamel mill liquor into the cover enamel. This defect is caused by an excessive concentration of the soluble salts. The second defect is the tearing of the cover enamel adjacent to the bead. These two defects will be referred to as "blistering" and "tearing."

Two types of enamels have been developed in recent years for one cover-coat kitchenware in which both defects may occur, namely (1) the antimony-free enamel, in which the frit is opacified with zirconium oxide, and (2) the acid-resistant enamel, in which the frit is opacified with antimony oxide.

Trials were made to determine the effect of milling and application variables on the dry-beading defects of both types of enamels.

II. Experimental

The trials extended over a considerable period of time during which several frits of each type were used. The results with a particular frit, in general, were applicable to the other frits in the same classification.

In contrast to frits opacified with antimony oxide, the antimony-free frits were distinguished by their high B_2O_3 and low alkali contents. Calculated analyses of the antimony-free frits indicated the following oxide limits:

	(%)		(%)
KNaO	10–12	SiO_2	37–39
CaO	3–5	ZrO_2	6–8
ZnO	2–4	F	10–12
B_2O_3	14–16	P_2O_5	1–2
Al_2O_3	7–8		

In contrast to nonacid-resistant frits, the acid-resistant types were characterized by their low B_2O_3 and Al_2O_3 and high alkali and SiO_2 contents. Calculated analyses of the acid-resistant frits indicated the following oxide limits:

	(%)		(%)
KNaO	17–19	TiO_2	4–6
CaO	1–2	Sb_2O_5	8–10
B_2O_3	6–9	F	6–7
Al_2O_3	2–6	P_2O_5	1–2
SiO_2	43–47		

In the discussion of the results, the antimony-free enamels and the acid-resistant enamels are listed as enamels A and B, respectively. The following basic mill formulas were used for these enamels:

	Enamel A	Enamel B
Antimony-free frit	100	
Acid-resistant frit		100
German Vallendar clay	7	
Ky. ball clay		7
Zirconium opacifier	6	4
Zinc oxide	2	
Magnesium carbonate	1/10	
Potassium carbonate		1/2
Water	40	38
Specific gravity	1.76–1.80	1.80–1.85

The enamels were ground in 1-gallon jar mills to a fineness of 12 gm. of dry residue on a 200-mesh screen.[1] Analyses for the Na_2O and B_2O_3 content of the mill liquors of the freshly milled enamels indicated the limits shown in the following table. The limits of a large number of mill-liquor analyses of nonacid-resistant enamels, in which the frits were opacified with antimony oxide, are also shown for comparison.

	Enamel		
Mg./10 cc.	A	B	Normal
Alkalis as Na_2O	6–8	40–45	12–16
B_2O_3	9–13	20–25	14–20

The enamels were adjusted to the best dipping consistency by the addition of potassium carbonate unless otherwise indicated. Flat-rim, ground-coated pans were dipped in one coat of 45 gm. per sq. ft., dry weight. The beads were sponged free of cover enamel before drying, after which the pans were beaded. Different types of beading enamels were used during the trials. The effect of variation in the beading enamel is given in the results. After the beading enamels were dried, the pans were fired for 2 1/2 minutes at 1560°F. in a laboratory electric box furnace.

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[1] Determined by the Standard Method of the Enamel Division of The American Ceramic Society.

III. Results

Both of the enamels, A and B, tore slightly at the bead under normal test conditions. Enamel A had a slightly blistered water line, and enamel B had a heavily blistered water line. Although there are seemingly a large number of methods of preventing these defects, most of them caused other defects which made their use objectionable.

(1) Clay Addition

(*A*) *Enamel A:* German Vallendar, purified German Vallendar, English ball, Kentucky ball, and several domestic enameling clays were compared. The ball clays had a slightly greater blistering tendency than the others, probably caused by the higher addition of potassium carbonate required to secure proper dipping consistency. The German Vallendar clay and one domestic clay from Mississippi were superior to the others in resistance to tearing at the bead.

(*B*) *Enamel B:* The Kentucky ball clay used is practically a standard for acid-resistant enamels because it imparts unexcelled dipping consistency and opacity. The various clays gave the same relative results as in enamel A.

The addition of $^1/_8$ to $^1/_4$% of bentonite had no effect on the blistering of either enamel. This addition was sufficient to eliminate the tearing at the bead. The use of bentonite, however, caused an objectionable gummy consistency.

An increase in the clay addition decreased the tearing in both enamels with no effect on the blistering.

(2) Opacifier Addition

All types of commercial opacifiers were compared in both enamels. The opacifier addition had no effect either on the blistering or on the tearing.

(3) Mill-Added Electrolytes

(*A*) *Enamel A:* The substitution of $^1/_{16}$ to $^1/_8$% of sodium nitrite for the $^1/_{16}$% of magnesium carbonate in the standard mill formula eliminated the blistering and decreased the tearing. The magnesium carbonate was not the cause of the blistering because it could be eliminated with no effect on the dry-beading defects. The addition of potassium carbonate at the dip tank was not necessary when sodium nitrite was used in the mill addition. Sodium nitrite eliminated the blistering even though potassium carbonate was added at the dip tank. The addition of $^1/_8$% of sodium nitrite eliminated the blisters. They reappeared, however, with additions above $^1/_4$%. High additions caused extreme blistering. The use of potassium carbonate in the mill addition increased the blistering.

(*B*) *Enamel B:* Potassium carbonate, which is practically the standard electrolyte for this type of enamel, unquestionably was the cause of the blistering. The substitution of practically any other common electrolyte for potassium carbonate eliminated the blisters. Sodium nitrite was an exception, being at least as bad as potassium carbonate. The use of Epsom salts, potash alum, ammonium carbonate, sodium aluminate, or magnesium carbonate resulted in inferior dipping consistency, decreased opacity, and increased tearing. Calcium chloride imparted fair dipping consistency to the enamel; the opacity, however, was greatly decreased. There are several commercial setting agents for acid-resistant enamels which are essentially hydrated aluminum silicates. The use of $^1/_2$ to 1% of these materials to replace the $^1/_2$% of potassium carbonate eliminated the blisters. Although satisfactory dipping consistency was secured with the setting agents, the opacity was decreased, and the enamels had considerable tendency to shoreline.

The addition of sodium nitrite to these enamels caused an increase in blistering contrary to the results secured with enamel A. A series of trials was made with other reducing agents to determine whether any would have the same effect in enamel B as sodium nitrite had in enamel A. The addition of hypo, sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$), was effective in the elimination of the blistering. Only 0.1 to 0.2% was required in the enamel containing 0.5% of potassium carbonate in the mill addition. The small addition of hypo had little effect on dipping consistency. No scumming difficulties were encountered with hypo additions below 0.5%. Several organic reducing agents decreased the blistering; each, however, had objectionable characteristics. Among these reducing agents were hydrazine sulfate and hydroxyl amine hydrochloride, which caused poor dipping consistency.

(4) Miscellaneous Mill Additions

Organic binders were tested in both enamels. The addition of up to $^1/_{16}$ to $^1/_8$% of gum arabic, gum tragacanth, casein glue, and several synthetic resins had no effect on tearing. These materials, however, caused an objectionable gummy consistency.

The addition of zinc oxide and titanium dioxide up to 2% had no effect on blistering or tearing.

(5) Fineness and Aging of Enamel

The tearing at the bead was greatly increased by finer grinding and was decreased by coarser grinding, which was objectionable in enamel A because it caused a mottled appearance and decreased opacity; in enamel B, coarser grinding decreased the acid resistance and opacity.

Blistering and tearing were increased by aging the milled enamel, and these effects were accentuated by aging at high temperatures.

(6) Dip Tank Electrolytes

(*A*) *Enamel A:* The effectiveness of sodium nitrite in the elimination of blistering and tearing was the same whether it was added in the mill or at the dip tank. The use of potassium carbonate in the dip tank increased the blistering.

(*B*) *Enamel B:* As the mill addition of potassium carbonate was varied from 0 to $^3/_4$%, the amount required at the dip tank was decreased. With $^3/_4$% in the mill, none was required in the dip tank. The addition of all necessary potassium carbonate to the mill caused more blistering than when it was added at the dip tank. The best dipping consistency, however

was obtained by adding all or practically all of the potassium carbonate in the mill.

(7) Application

Blistering and tearing were increased by a heavier application of the cover enamel. Poor sponging, which built up a heavy layer of cover enamel, also increased these defects

(8) Beading Enamel Composition

Variation in the beading enamel, except in its electrolytes, had little effect on blistering and tearing. Blistering was decreased in enamel A by the use of $^1/_{16}$ to $^1/_8$% of sodium nitrite in the beading enamel; under certain conditions, however, sodium nitrite caused a discoloration in red beading enamels. Blistering was increased in both enamels by the use of potassium carbonate in the beading enamel.

The soluble salts in the cover enamel had more influence on the extent of blistering than did those in the beading enamel. Distilled water was allowed to penetrate the cover enamel in the same manner as the mill liquor from the beading enamel. There was practically no difference in the resultant amount of blistering.

Blistering was completely eliminated in both enamels by using an organic solvent, such as alcohol, for the carrier in the beading enamel. The soluble salts in the cover enamel were insoluble in the alcohol and therefore were not concentrated at its line of deepest penetration. The beading enamel in which alcohol was used did not have entirely satisfactory consistency. Alcohol is objectionable because of its high cost and because of fire and health hazards.

Variation in the hydrogen-ion concentration of the beading enamel had no effect on the dry-beading defects. Enamel A, at a pH of 8.6, was used in this trial. The pH of a beading enamel was varied from 9.7 to 8.6 and from 9.7 to 10.7 by the addition of hydrochloric acid and sodium hydroxide, respectively, with no effect on blistering and tearing.

(9) Beading Application and Firing

The dry-beading defects were kept at a minimum by light application of the beading enamel. Overlapping of the cover enamel by the beading enamel greatly increased both defects. Rapid drying, after the beading enamel was applied, decreased the penetration of the mill liquor and therefore decreased the blistering.

The blistering in the water line was greatly increased by longer firing or higher temperatures.

IV. Conclusions

(1) The extent of blistering is controlled by the quantity and nature of soluble salts deposited in the water line. (2) The quantity of soluble salts deposited in the water line may be kept at a minimum by careful application of the cover and beading enamels. (3) The most common electrolytes have little effect on blistering; a concentration, however, of potassium carbonate in the water line causes blisters. (4) Under certain conditions, small additions of sodium nitrite or sodium thiosulfate to the cover enamel are effective in eliminating blisters. They are also effective, to a lesser degree, when they are added to the beading enamel. (5) Tearing at the bead is influenced by the same factors that govern the usual tearing in all enamels.

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EFFECT OF ISOMORPHOUS SUBSTANCES ON GLASS*

BY ALEXANDER SILVERMAN

The aluminum atom will displace the silicon atom in silicon-oxygen tetrahedra in certain silicates and glasses. Alumina is in the hexagonal system to which quartzite belongs, and quartzite has been recognized in the crystalline phase of opal glasses. If metallic oxides are replaced by alumina in the glass batch, the increase in concentration of aluminum may cause a further separation of quartzite crystals and perhaps also of aluminum oxide crystals. In any event, increased concentration of alumina, whether to displace silica or metallic oxides, increases the opacity of opal glasses.

Aluminum phosphate is also hexagonal, which may account for the great opacity produced by phosphates in glass batches. Beryllium oxide crystallizes hexagonally, and it will be interesting to see how this compound affects opal glasses. The red color in selenium glasses is said to be due to tiny crystallites of cadmium selenosulfide. A study of the influence of substances isomorphous with this may also prove to be interesting.

On the basis of practical results already seen and general trends in science studies, the theory is advanced that isomorphous substances will either increase crystallization in a glass in which similarly crystallized materials are present or they will induce crystallization where this has not yet taken place.

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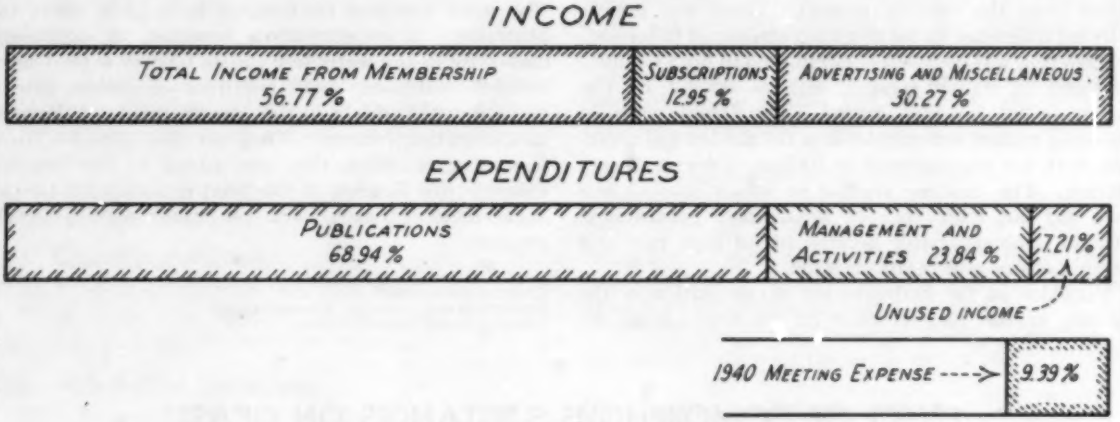

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E. W. W. Bartlett	J. L. Gallup	G. M. Hutt	W. J. Leszynski	M. E. Poor	F. E. Sonnen
A. C. Bevan	J. D. Gat	J. F. Hyde	W. Loewenthal	Katherine Reed	E. Stefanowski
D. A. Biddle	R. A. Gregory	Herbert Insley	F. S. Mallette	H. K. Richardson	A. G. Stern
A. Bidduiph	Robert Halle	C. B. Jenni	V. S. de Marchi	R. A. Ronnebeck	B. H. Strom
P. P. Budnikov	Max Hartenheim	B. Z. Kamich	A. F. Matthews	B. C. Ruprecht	M. Takei
J. C. Chaston	J. J. Hazel	B. E. Kinkulkin	L. F. May	A. B. Searle	L. E. Thiess
W. M. Cohn	F. G. Heck	G. A. Kirkendale	E. H. McClelland	Stanford Setchell	L. W. Vermeulen
M. V. Condoide	R. A. Heindl	Yosio Kora	J. M.Noy	Lucille Shattuck	F. J. Zvanut

Journal Reference: The journal name appears in italics, followed by the volume (bold-faced), issue number (in brackets), page numbers, and year (in parentheses).

July, 1941
20—7

Abrasives

Abrasive blast cleaner reduces burr-removal costs. Anon. *Iron Age*, **147** [16] 50 (1941).—When the cost of grinding is prohibitive, burrs on machined parts can be successfully and inexpensively removed by airless blasting with metallic grit. The treatment also provides an excellent bond for subsequent surface coating. E.H.McC.

Dust collector for tool grinder. Anon. *Iron Age*, **145** [21] 55 (1940).—With a Carboloy grinder, dust is taken care of by an installation costing $110 for labor and equipment. A small blower is driven by a $\frac{1}{2}$-h.p. motor. The intake side of the blower is connected to a sheet-metal chamber; a pair of 2-in. feeder lines runs from the hood of the grinder to the chamber. The output side of the blower connects with a 4-in. sheet-metal pipe which conveys the dust to a long canvas bag. The bag can be opened at the bottom to remove accumulated dust. E.H.McC.

50th anniversary of manufactured abrasives. Francis D. Bowman. *Iron Age*, **147** [10] 56–57 (1941).—The history of E. G. Acheson's Carborundum is outlined; brief comments are given on the importance of silicon carbide and aluminum oxide. E.H.McC.

Finishing dies and molds. H. J. Wills. *Iron Age*, **146** [12] 54–55 (1940).—A die roughly shaped by cutting tools is brought to correct shape, dimensions, and finish by means of abrasives. All stages of finishing may be accomplished either manually or by a flexible-shaft grinder. The procedures discussed include rough polishing, fine polishing, and buffing. F.H.McC.

Grading abrasive sticks by the Rockwell test. Lowell H. Milligan. *Iron Age*, **146** [12] 50–52, 102 (1940).—The Rockwell hardness-testing machine is widely available. When carefully used under the limitations indicated, it affords a quick and easy method which is of value for determining the bonding strength of fine vitrified abrasive products of relatively soft grade. The Rockwell test, however, does not give any information which is new or different from that obtainable by other grading tests conducted by the Norton Co. Comparative results of tests are given. See *Bull. Amer. Ceram. Soc.*, **20** [2] 39–47 (1941). E.H.McC.

Grinding and sanding castings without dust. Anon. *Iron Age*, **146** [11] 51 (1940).—At the plant of the Monarch Machine Tool Co., the top of each working station is a large grating through which air is exhausted (through the roof) at an average surface velocity of 200 ft. per min. Castings are now ground, sanded, and spray painted without partitions to separate the operations. E.H.McC.

New abrasive produced by crystallization cementation. Anon. *Ceramica*, **2** [12] 466 (1940).—The new abrasive disks, Aldemite-Cristallo (Società Italiana dello Smeriglio), contain a lower percentage of agglomerant than ceramic processes usually permit and a higher percentage of the abrasive element. This is obtained by a cementation of the abrasive granules by means of crystallization. The abrasive has a longer life, cuts better, and holds the cutting angle better. It does not heat up as rapidly during grinding and is safer to use. M.V.C.

Separation of fine abrasive powder by means of electric precipitation using ionized steam. Y. Toriyama and K. Kurihara. *Electroteck. Jour.* [Japan], **4** [9] 216 (1940).—By the use of ionized steam, the efficiency of electric precipitation is increased compared with that of the usual Cottrell precipitators. Powders with diameters of less than 7 μ can be precipitated and may be used as abrasive powders for optical lenses. W.J.L.

Superfinish improved by abrasive uniformity. W. F. Sherman. *Iron Age*, **147** [20] 46–47 (1941).—Abrasive stones are frequently graded by hardness, but either the Rockwell or the Brinell values can be misleading if the grain structure is not thoroughly uniform. One solution of this problem is the use of a "neutralizing" treatment by which each grain is isolated and prevented from adhering to adjoining grains. Treated grits, in the unbonded state, are of high capillarity, and the bonding agent is finer than the material being bonded so that complete coating of each grain of abrasive is achieved. This bonding is called "microbond." A new method of rating stones

is based on the hardness of the bond and on the density of the stone, the latter being related to the controlled pressure used in manufacturing the stones. See "Conception . . . ," *Ceram. Abs.*, **20** [6] 135 (1941). E.H.McC.

PATENTS

Abrasive article and method of making. A. W. WALKER (Carborundum Co.). U. S. 2,241,433, May 13, 1941 (Dec. 11, 1939).—An abrasive grinding disk.

Abrasive disk and method of making. F. O. ALBERTSON (Albertson & Co., Inc.). U. S. 2,242,877, May 20, 1941 (March 15, 1939).

Abrasive tool. J. N. BETHEL (Norton Co.). U. S. 21,789, May 6, 1941 (July 20, 1940); reissue of original U. S. 1,931,370, Oct. 17, 1933 (Nov. 2, 1928).—An abrasive tool comprises a molded, compacted mixture of separate and distinct particles of granular metal and granular abrasive material held in position by a brittle, nonmetallic bond, which has been heat-set to form an integral body.

Apparatus for honing internal screw threads. J. H. HOERN (Eaton Mfg. Co.). U. S. 2,240,440, April 29, 1941 (Dec. 5, 1940).

Automatic infeed mechanism for grinding machines. JACOB DECKER (Cincinnati Grinders, Inc.). U. S. 2,241,634, May 13, 1941 (April 12, 1940).

Automatically expansible polishing and buffing wheel. H. J. BARTON AND R. H. BARTON (F. L. Szafranski and C. E. Wisner). U. S. 2,239,140, April 22, 1941 (Jan. 2, 1940).

Axially slidable supporting means for grinding wheels. LIDKÖPINGS MEKANISKA VERKSTADS AKTIEBOLAG. Brit. 535,481, April 23, 1941 (Oct. 31, 1938).

Buffing device. P. E. HAWKINSON (P. E. Hawkinson Co.). U. S. 2,240,559, May 6, 1941 (Dec. 4, 1939).

Coated abrasive. N. E. OGLESBY (Behr-Manning Corp.). U. S. 2,239,828, April 29, 1941 (Feb. 6, 1937).

Composition for impregnating grinding wheels. H. H. JONES. U. S. 2,240,302, April 29, 1941 (Sept. 12, 1940).—An impregnating composition for cutting wheels comprises hydrogenated cottonseed oil 90 to 97, salt 0.75 to 7.5, sulfur 0.75 to 7.5, and a penetrating agent 0.75 to 7.5%.

Crankshaft grinder. JOSEPH SUNNEN. U. S. 2,240,527, May 6, 1941 (May 22, 1939).

Crankshaft grinding machine. C. G. FLYGARE (Norton Co.). U. S. 2,241,342, May 6, 1941 (Nov. 22, 1940).

Cutter and tool-grinding machine. C. H. AMIDON (Norton Co.). U. S. 2,239,639, April 22, 1941 (March 23, 1940).

Grinding machine. C. G. FLYGARE (Norton Co.). U. S. 2,239,091, April 22, 1941 (April 5, 1939). V. W. GIDEON (Boyar-Schultz Corp.). U. S. 2,242,781, May 20, 1941 (Jan. 24, 1940).

Grinding wheel. S. S. KISTLER AND C. E. BARNES (Norton Co.). U. S. 2,243,049, May 20, 1941 (Aug. 19, 1939).—An abrasive body comprises abrasive grains, a bond holding the abrasive grains together, there being pore spaces in the wheel, and a finely divided solid compound which is strongly acid in reaction at grinding temperatures above 500°C., uncombined with the abrasive or bond, available for reaction with a metal workpiece during grinding when the grinding line gives off sparks, i.e., sparks above 500°C. in temperature, and selected from the group consisting of the anhydrides and acid salts of sulfuric and phosphoric acids.

Grinding wheel dressing fixture. R. E. CROSS. U. S. 2,239,034, April 22, 1941 (Feb. 12, 1940).

Headstock for machine tools, particularly grinding machines. KARL JUNG. U. S. 2,239,103, April 22, 1941 (June 28, 1938).

Knife sharpener. A. E. MEADE AND M. L. MEADE. U. S. 2,241,412, May 13, 1941 (Aug. 9, 1939).

Lapping machine. H. S. INDGE AND F. E. JOHNSON (Norton Co.). U. S. 2,241,351, May 6, 1941 (April 15, 1940).

Machine for finishing the surface of a workpiece by abrasion. CINCINNATI GRINDERS, INC. Brit. 535,485, April 23, 1941 (July 31, 1939).

Machine for grinding bodies of rotation with curved generatrices. E. G. HJÄRPE (Aktiebolaget Svenska Kullagerfabriken). U. S. 2,242,031, May 13, 1941 (March 28, 1939).

Machine for sharpening rotary cylindrical cutters. M. C. MCCORMACK. U. S. 2,239,683, April 29, 1941 (Feb. 7, 1940).

Stone grinding or polishing wheel. WILLIAM MILNE. U. S. 2,242,753, May 20, 1941 (July 24, 1940).

Washer grinding machine. A. T. RICHARDSON (Woodings-Verona Tool Works). U. S. 2,242,801, May 20, 1941 (Feb. 21, 1940).

Work-gauging instruments for precision grinding machines. CHURCHILL MACHINE TOOL CO., LTD., AND H. H. ASBRIDGE. Brit. 535,503, April 23, 1941 (March 21, 1940).

Art and Archeology

Amusing ceramic art. ANON. *Design*, **42** [8] 16–17 (1941).—The fact that art can be amusing is often overlooked. Too often the impression exists that art must always be serious. A sense of humor is a valid and commendable thing to maintain in all forms of art expression. Eight illustrations of humorous pieces of ceramic sculpture by different artists are shown. L.S.

Belle Vue Pottery, Hull. T. SHEPPARD. *Trans. Brit. Ceram. Soc.*, **40** [1] 1–13 (1941).—A historical presentation covering styles and body compositions is given. R.A.H.

Ceramic art in Cleveland public schools. ALICE A. AYARS. *Design*, **41** [8] 14–16 (1940).—Increased emphasis is being given to the art of the potter. The part played by the public school art departments in bringing about this condition is described. Approximately thirty years ago a deep interest in pottery was fostered by several of the art teachers; this interest has steadily increased until, today, clay work is taught by nearly every art teacher at some time during the semester. The public schools have been quietly laying the groundwork for the present so-called ceramic consciousness so evident in the city of Cleveland. The influence of the public school art teachers in raising the level of appreciation and of artistic production cannot be overestimated. L.S.

Ceramic sculpture created in one of the secondary schools of Cleveland under the direction of an artist-teacher, Thelma Frazier. *Design*, **41** [2] 23 (1939).—Photographs of ceramic sculpture by children of secondary school age are presented. L.S.

Clay plaques in school publications. OMA STRAIN. *Design*, **41** [1] 12 (1939).—The pupils of Norwood High School, Norwood, Ohio, produced interesting division pages for their school publication by using photographs of clay plaques which they had modeled. The advantages and disadvantages of the project are listed. Several desirable effects are noted, including the increased interest shown in the modeling classes. L.S.

Copper luster bowls. ANON. *Design*, **40** [9] 12 (1939).—The application of copper luster to common earthenware already fired is explained and recommended as an introduction to the potter's craft. L.S.

Creative modeling. ANON. *Design*, **42** [1] 8–13 (1940).—Photographs of ceramic sculpture from Herbert Hoover High School, Glendale, Calif., made under the direction of Gladys D. Merrick, are presented. L.S.

Decentralization and art education—pottery. MINNIE MCLEISH. *Studio*, **121** [574] 12–15 (1941).—The fascination of making pots by throwing and by simpler means, such as coiling pencil-thin rolls of plastic clay, is introduced to beginners, especially children. Illustrated. L.R.B.

Figurines of Naples. G. MORAZZONI. *Ceramica*, **2** [12] 441–56 (1940).—The art of making Neopolitan figurines originated toward the end of the 17th century with the

work of Antonio Vaccaro who started the vogue for the sculptured and painted statuette. Originally the figures were carved from wood; later they were composed of three materials: the bust was of painted terra cotta, the body (modeled over a wire framework) was clothed in the dress materials of the time, and the hands and lower extremities were of carved, painted wood. There was an infinite variety of types, e.g., religious characters, rustics, and artisans at work in very lifelike and natural poses. The features are remarkably expressive and detailed. The figurines furnish a historical document of the life and dress during the reign of Carlo III and Ferdinand IV. They were made by true ceramic sculptor-painters who had a thorough knowledge of anatomy; outstanding among the artists were Giuseppe Sammartino, called the Donatello of figurines, Mosca, Gori, Celebrano, the Bottiglieri brothers, Somma, and Viva. Excellent photographs of some of the figures are given. M.V.C.

Graphic arts for ceramic decoration. HANS FREY. *Sprechsaal*, **72** [15] 186–87 (1939).—The old methods of copper and steel engraving combined with hand painting for decorating porcelain have been replaced by mechanical color printing. A new process offering many possibilities in the decoration of porcelain and ceramic ware is the "photo-hand-copperplating" process for the authentic reproduction of pictures or paintings. It is especially suitable for decorating with cobalt and may be used on porcelain and on whiteware underglaze in one or more colors. The copper plates can be made to last longer by galvanizing with a steel coat. Hand etching with its technical combinations has been used with fine results by F. for reproducing landscapes, figures, flowers, etc. The hardest steel was used. M.V.C.

From the Saxon Electorate Art Gallery to the Zwinger State Porcelain Gallery: III. F. FICHTNER. *Ber. Deut. Keram. Ges.*, **21** [9] 330–70 (1940).—F. covers the history of the Dresden collection of porcelain from 1833 to 1933. For Parts I and II see "Dresden . . . ," *Ceram. Abs.*, **17** [11] 347 (1938). W.H.H.

Medieval heraldic glass in Surrey churches: III. F. SYDNEY EDEN. *Connoisseur*, **107** [473] 28–31 (1941).—E. describes 14th and 15th century heraldic glass in churches at Witley and Worplesdon. Much of the ancient glass originally located at Surrey has been thrown away as rubbish, is in private collections, or has been removed to the U. S. Much of it was destroyed during the religious upheavals of the 16th and 17th centuries. Illustrated. For Part II see *Ceram. Abs.*, **20** [5] 111 (1941). M.E.P.

Modeling done at the Music and Art Foundation, Seattle. *Design*, **41** [2] 15 (1939).—Photographs of the work of Pat Sullivan are presented. L.S.

Modern Italian ceramics. *Design*, **41** [2] 11 (1939).—Nine photographs of modern Italian ceramics showing different decorative processes are presented. L.S.

Origin of porcelain in China. R. LECUIR. *Compt. Rend.*, **211** [13] 262–64 (1940).—Vitrified and translucent ceramics were made in China during the Sung dynasty in about the 10th century; their manufacture in Europe, however, is fairly recent. Earlier, under the Tang dynasty, Chinese ceramics were made on a kaolin base but were fired at a temperature which would give only a hard and opaque ware, sometimes porous and not translucent. Later the temperature was increased to 1200° or more to produce translucent porcelain. The compositions of old and modern Chinese porcelains and of European porcelain are practically identical, as shown in the following table:

	SiO_2	Al_2O_3	Fe_2O_3	CaO	TiO_2	MgO
China (Sung)	71.66	21.11	1.41	1.18	0.20	0.44
China (19th century)	70.50	20.70	0.80	0.50		0.10
European	71.20	22.00	0.80	0.80		

Although kaolin porcelains require a firing temperature of about 1300°C. (according to modern practice), the Chinese apparently knew how to adapt their technique to compensate for the low firing temperature. M.H.

Pinnacle of French rococo porcelain. MARVIN CHAUNCEY ROSS. *Art News*, **40** [4] 26 (1941).—The *pâte tendre* of Sèvres set the standard of taste for other factories in the 18th century when it was patronized by Louis XV, Mme. de Pompadour, Louis XVI, Mme. Du Barry, the Duc d'Aumont, the Princess de Talmont, and, just before the French Revolution, Marie Antoinette. The Walters Art Gallery's public exhibition of its great collection of soft-paste porcelain celebrated the annual meeting of the American Ceramic Society, Baltimore, 1941. This porcelain was first made in Rouen, France, a hundred years after its discovery at the Medici court in Florence. It was very white and opaque because of the tin glaze. It took glazes beautifully, better than hard-paste porcelain. Factories at St. Cloud and Chantilly had long been established when, in 1738, the Dubois brothers persuaded Orry de Fulvy to start a factory at Vincennes which began to succeed in 1745 when it was transferred to François Gravant. In 1753, Louis XV became one-fourth share holder, and it was called *Manufacture royale de porcelaine de France*, two interlaced L's being its mark. It was moved to new quarters at Sèvres in 1759, and Louis XV took it over; it remained in royal hands until the Revolution. When silver was melted down to bolster French finances, Sèvres porcelain became even more popular, as it replaced silver tableware. The factory continued making soft-paste porcelain until kaolin was found at St. Yrieix near Limoges in 1768. By 1772, a hard-paste process was perfected, and, in 1800, Brongniart put the business on a successful financial basis. A special quality of gold and lovely ground colors are distinctive of Vincennes and Sèvres soft-paste porcelain. Descriptions and illustrations of outstanding examples in the Walters collection are given. M.E.P.

Primavera ware. *Design*, **41** [3] 19 (1939).—Photographs of Primavera ware from Primavera, including tableware made for Printemps, Paris, are presented. L.S.

Role of the engobe in ceramics of the Far East. R. LECUIR. *Compt. Rend.*, **211** [16] 328–30 (1940).—In general, an engobe is defined as a paste which is put on the unglazed material, either to hide the color of the latter or as decoration. In L.'s investigations of old Chinese ceramics, however, he found that the engobe was used for a different purpose from about the 10th century (Sung dynasty). The engobe was vitrified while the body was still too incompletely fired to be perfectly impermeable. The engobe generally is covered with a coat of a transparent or opaque glaze. The crackling of this glaze is not a defect caused by or in the manufacture but is brought about by the vitreousness of the engobe which it covers. This crackled appearance is characteristic of this epoch. The application of this engobe was gradually abandoned as the means of firing were perfected, but it persisted as a means of decoration up to the 17th century. M.H.

Stained glass. MURIEL DEY. *Design*, **40** [8] 8–15 (1939).—A summary of glassmaking in ancient Egypt is followed by a review of Roman glass and mosaics. The process of making stained glass is fully described, special emphasis being given to stained glass of the 12th century. Photographs of the principal steps in the process of making stained glass are shown. L.S.

Use of native materials in rural schools. FELIX PAYANT. *Design*, **41** [10] 13–14 (1940).—An awareness of native materials in the community has been associated with the development of civilization from the very beginning. Instead of having less opportunity in art than the highly compartmentalized school systems of the larger cities, the teacher in the one-room school or the consolidated school has a decided advantage over teachers in city schools. The use of native clays for pottery making, for modeling, and for making show-card colors and finger paint is suggested. The great argument in favor of native materials is that a valuable point in art appreciation is the understanding of how man made his living vivid, interesting, and beautiful by converting natural resources into the many fine things we see about us today. L.S.

Carl Walters. *Design*, **41** [2] 16 (1939); *Art News*, **38** [12] 10 (1939).—Photographs of the sculptor, Carl Walters, and his work are presented. L.S. + M.E.P.

BOOKS

Outline of Modern Chromatics. MANFRED RICHTER WITH INGEBORG SCHMIDT AND ALBERT DRESLER. Theodor Steinkopff, Dresden, Leipzig, 1940. 226 pp., 120 illustrations, 18 tables. Price Rm. 11.25; bound, Rm. 12.38.—A comprehensive introduction to the theories, principles, and practical application of the science of color as it has been advanced by the latest research is presented. It is of value to scientists, technicians, engineers, opthalmologists, physiologists, psychologists, teachers, and students and in all branches of industry where colorimetry and the characterizing and measuring of color are important. Chapter headings are as follows: (*A*) Fundamental conception of color; (*B*) Laws of additive color mixing; (*C*) Brightness of colors; (*D*) Physics of color; (*E*) Spectrum measurement; (*F*) Physiology of color seeing, by Ingeborg Schmidt; (*G*) Pathology and theory of color seeing, by I. Schmidt; (*H*) Color opposites; (*I*) Color stimulus; (*J*) Colorimetry and apparatus; (*K*) Color sensitivity; (*L*) Heterochromic photometry, by Albert Dresler; (*M*) Color lighting; (*N*) Application of chromatics and colorimetry. An appendix, bibliography, and an index are included. M.V.C.

Pottery Making from the Ground Up. YORK HONORÉ. Viking Press, New York, 1941. 72 pp., 44 reproductions from photographs by Larry Morris, Jr., 11 working drawings. Price $1.00.—Within a mile or two of almost anyone's house there is clay to be had for the digging, from which even a beginner, guided by the clear photographs and the explicit text, can fashion pitchers and other pottery and even a kiln in which to fire and glaze them. The following points are covered: choice of a suitable clay, preparation for use, position of the hands in shaping the clay, the building of a kiln "from the ground up," home construction and use of a potter's wheel, etc. This is a unique craft book about a hobby to which no one is immune.

York Honoré is potter, craftsman, teacher of art, and general handyman. He says of himself: "I hate to admit that I can't do all of any job I attempt . . . and, as I now live on a farm, this attitude keeps me considerably occupied. I was born in 1912 in Detroit. In college I tried to make my way as a commercial photographer but found that I was not (and am not) a businessman. Then I got a job teaching pottery and found I had a bent for it. My things sold, so I went further with it and studied chemistry and sculpture."

The Honoré homestead is on the Susquehanna River in Maryland and is the scene of endless experiments in all kinds of media, e.g., pottery, painting, photography, and even the construction of rammed-earth houses.

PATENTS

Designs for:

Base for poultry fount and feeder. M. T. BENTZEN AND J. F. NIGGELER. U. S. 126,997, May 6, 1941 (Feb. 7, 1941).
Book end. CARL SCHMITZ (Verlys of America, Inc.). U. S. 127,311, May 20, 1941 (March 11, 1941).
Bottle. J. DE M. VICENT (J. J. Blanco). U. S. 126,725, April 22, 1941 (Dec. 16, 1940).
Bowl. CARL SCHMITZ (Verlys of America, Inc.). U. S. 127,312, May 20, 1941 (March 11, 1941).
Comport. C. W. GUSTKEY (Imperial Glass Corp.). U. S. 127,271, May 20, 1941 (March 25, 1941).
Goblet. C. W. CARLSON. U. S. 127,154, May 13, 1941 (March 4, 1941).
Jar. H. M. GEHM. U. S. 126,977, May 6, 1941 (March 24, 1941).
Lavatory. H. V. KOHLER AND C. W. ZILLIER. U. S. 127,274, May 20, 1941 (March 24, 1941).
Lighting fixture. T. W. ROLPH (Holophane Co., Inc.). U. S. 126,750, April 22, 1941 (June 26, 1940).
Line insulator. L. J. SCHILLING (Babson Bros. Co.). U. S. 126,722, April 22, 1941 (Aug. 2, 1940).
Milk bottle. ARTHUR BRAMHALL. U. S. 126,711, April 22, 1941 (Jan. 2, 1941).
Plate. ISOBEL DARROW (Pacific Clay Products). U. S. 126,975 and 126,976, May 6, 1941 (May 1, 1940). C. W. LEIGH (Morimura Bros., Inc.). U. S. 127,297 to 127,300, May 20, 1941 (March 29, 1941). U. S. 127,352, May 20, 1941 (March 29, 1941). M. C. RUSKIN. U. S. 127,075 to 127,077, May 6, 1941 (Oct. 28, 1940).
Stemmed goblet. W. P. MORRISON (Bryce Bros. Co.). U. S. 126,837, April 29, 1941 (March 10, 1941).
Strain insulator. L. J. SCHILLING (Babson Bros. Co.). U. S. 126,721, April 22, 1941 (Aug. 2, 1940).
Vase. CARL SCHMITZ (Verlys of America, Inc.). U. S. 127,309 and 127,310, May 20, 1941 (March 11, 1941).
Wine bottle. R. H. DALLAS (Glass Containers, Inc.). U. S. 127,007, May 6, 1941 (Dec. 16, 1940).

Cements

Effect of TiO_2 on the properties of Portland cement. L. D. ERSHOV. *Prom. Stroitel. Materialov*, **2** [10–11] 29–33 (1940).—The introduction of not more than 5% TiO_2 into the batch has no visible effect on the properties of Portland cement but influences its physicochemical properties somewhat favorably. TiO_2 in an amount exceeding 5% has a detrimental effect on the properties of cement. In the calcining process, the calcium compounds of TiO_2 are included in the composition of basic clinker calcium silicates, improving their optical characteristics. With 3 to 5% of TiO_2, no improvement in the optical characteristics of alite and belite is observed. With 8 to 10% TiO_2, there is a considerable improvement in these characteristics and a change in the hydraulic and physicotechnical properties, resulting in poor hydraulicity and poor physicomechanical characteristics. P.B. & E.S.

Resistance of Portland cements of different mineralogical composition to the effect of sulfates. S. L. VOLFSON, S. D. OKOROKOV, AND T. N. BURAKOVA. *Prom. Stroitel. Materialov*, **2** [10–11] 34–37 (1940).—The resistance of Portland cements to the effect of sulfates can be changed to a great extent by changing their mineralogical composition; the greatest resistance is evidenced by cements containing a small amount of tricalcium silicate and no calcium aluminate. P.B. & E.S.

SEPARATE PUBLICATIONS

Portland Cements for Decoration—Their Properties and Uses. S. S. CHEREPOVSKIĬ AND O. K. ALESHINA. *Giprotsement*, No. 24, 35 pp. (1938).—The production, uses, and properties of colored Portland cements used for decorating purposes are described. M.V.C.

Reaction Between Calcined Kaolin and Lime in Aqueous Solution. W. STRAETLING. Thesis, Tech. Hochschule Braunschweig, 1937; reviewed in *Ber. Deut. Keram. Ges.*, **20** [12] 522–27 (1939).—S. believes that previous attempts to explain the chemical interaction between pozzuolana and lime have failed because the more complicated trass was used. A standard kaolin, therefore, was calcined at various temperatures, and the identification of the reaction products formed from the calcined lime and limewater was then undertaken. The higher the temperature at which the kaolin was calcined, the coarser were the particles, but the change was much slower above 500°C. If the heat of wetting, as found with water, is subtracted from the heat due to reaction with limewater, the difference is equal to the heat of adsorption. Budnikov ("Reaction . . . ," *Ceram. Abs.*, **16** [1] 7 (1937)) concluded from such tests that pozzuolana had the highest chemical activity with $Ca(OH)_2$ at 800°C. S., however, found for these heats (at 500°C.) values of 1.02, 1.71, and 0.69 cal.

per gm., respectively; these are the highest values he obtained for kaolin heated to any temperature. Shaking the calcined kaolins with limewater for periods varying from 111 to 1536 hr. gave the following results: (1) Kaolin calcined at 110° adsorbed CaO at first and showed very slight power of combination. (2) Kaolin calcined at 500° to 700° showed strong reactions; it is therefore especially suitable as a component of a mixed cement. (3) Kaolins calcined at higher temperatures showed a decrease in their ability to form compounds with lime. As systematic investigations of the system CaO–Al_2O_3–SiO_2–H_2O were lacking, S. made microscopic and X-ray examinations of the solid phase resulting from the reaction of the kaolin and lime and of the mother liquor. Calcium hydrate crystals were mainly observed. A comparison of the solid phase with natural and synthetic calcium silicate and calcium aluminate hydrate, as well as natural zeolite, showed that in no case were the new products identical with the others. The shaking test of the monohydrate of dicalcium silicate and the hexahydrate of tricalcium aluminate and the reaction between tricalcium silicate and tricalcium aluminate showed that, in aqueous solution, a reaction between calcium silicate and calcium aluminate resulted and that the reaction products were identical with the new products formed between kaolin and limewater. W.H.H.

PATENTS

Firing rotary kilns in the production of Portland cement. H. M. Armel. U. S. 2,240,485, May 6, 1941 (May 31, 1939).

Water-glass cement powder and preparation of cement mortar therefrom. Karl Dietz (Pen-Chlor, Inc.). U. S. 2,240,393, April 29, 1941 (Feb. 4, 1939).—The process of preparing a water-glass cement resistant to water and acids from a cement powder comprises mixing water with a cement powder which contains pulverulent water glass, an acid-resisting filling agent, and a substance capable of reacting with alkali, the pulverulent water glass having a ratio of SiO_2 to alkali oxide exceeding 2:1 and there being present in the cement powder a large excess of the substance capable of reacting with alkali over the quantity necessary for neutralizing the alkali in the water glass used.

Enamel

Analysis of clay. E. O. Primbsch. *Emailwaren-Ind.*, **18** [3–4] 7–8 (1941).—In rating a clay to be used for enameling purposes, it is necessary to know the water content, the Al_2O_3 and SiO_2 contents, and a few impurities which may affect the properties of enamels, e.g., iron, manganese, titanium, alkaline earths, and magnesium compounds. The steps in the analysis of clay are given. See *Ceram. Abs.*, **20** [4] 83 (1941). M.V.C.

De-enameling of sheet-iron parts. W. C. Cummings. *Jour. Can. Ceram. Soc.*, **9**, 73–75 (1940).—C. describes some experiences encountered in the de-enameling of sheet-iron stove parts using the caustic soda solution method. De-enameling brings to light poorly designed parts or parts on which losses are incurred, reclaims parts on which much labor has been expended in fabricating, welding, etc., and saves setting up dies if only a few parts are required or if the finish has to be changed. Under present-day conditions it is quite possible that steel prices will substantially increase; in this event there will be more economy in de-enameling than there has been in the past. With a tendency toward more welding and increased fabrication costs on stove parts, de-enameling of these parts has become increasingly important. J.G.P.

Enamel Division Standards Committee Report. Clark Hutchison, *Chairman*. *Bull. Amer Ceram. Soc.*, **20** [5] 176 (1941).

Hairlining of sheet-steel enamels. B. J. Sweo. *Jour. Can. Ceram. Soc.*, **9**, 63–72 (1940); see *Ceram. Abs.*, **19** [7] 158 (1940). J.G.P.

Majolica enamels free from lead and boron. H. Lang. *Glashütte*, **70** [52] 669–70 (1940).—In experimenting with the production of majolica enamels without lead, borax, or boric acid, L. worked out the following recipe which gives satisfactory results by the dry or powder method and which may serve as a basis for similar batches: glass powder (transparent) 40, quartz 12, barium carbonate 14, soda 16, zinc oxide 6, fluorspar 7, and saltpeter 5; for blue, 1% cobalt oxide. The high powdered-glass content gives this enamel a resistance and stability to corrosion which the usual majolica enamels seldom possess. Zinc oxide gives a good gloss; it may be replaced by barium carbonate. Unfortunately, not more than 7% fluorspar can be used because of its harmful effect on coloring oxides and its tendency to opacify. This majolica enamel succeeds best when the color oxide content can be kept low. When smelted at 800°, the powdered enamel is liquid and has a fine gloss, a good color (blue), and a good majolica character. It is, however, slightly less transparent; for this reason it must be applied very thin. M.V.C.

Pickling aids in enameling plants. Anon. *Glashütte*, **71** [4] 41–42 (1941).—Where little or no boric acid is to be used in the enamel, greater care must be exercised in pickling the metal before enameling because boric acid has the capacity to nullify the action of impurities in the surface of the metal which would otherwise produce defects in the enamel. The use of inhibitors in the pickling bath is especially advantageous in this case because they check the dissolving of the iron and the liberation of hydrogen, thus reducing the consumption of acid and the loss of metal. Pickling additions are divided into three groups according to their chemical structure: (1) chemical compounds and mixtures (hydrocarbons and sulfoacids of hydrocarbons); (2) natural and technical products of more or less known composition (3-membered heterocycles and 5-membered heterocycles with 1 to 3 hetero atoms); and (3) natural and technical products of more or less exactly defined composition (products from mineral oils, fats, and carbohydrates). Flaws in enamels caused by salt impurities in the surface of the metal can also be reduced by the hot Feracid treatment after pickling. The nickel bath after pickling is also recommended. M.V.C.

Preparing and enameling cast iron. Hugo Schulze-Rudniki. *Glashütte*, **71** [1] 6 (1941).—The recurrence of flaws in large enameled cast-iron objects, such as autoclaves, tanks, bathtubs, boilers, etc., can be cut down by more careful inspection and checking of the articles before enameling. Few plants have facilities for pickling such large objects; instead, sandblasting and polishing machines are used for cleaning the metal surface to remove particles of slag, sand, graphite, coal, and dirt. These machines must be inspected regularly to see that they are in proper running order and that the right pressure is maintained (6 atm. is best for sandblasting). Each article must be examined carefully, smooth places should be roughened, cold welding and tension cracks should be removed, and bubbles should be smoothed or riveted before enameling. It is advisable to keep a control book to record all defects which will cause flaws in the enamel, e.g., variations in the weight of the articles and in the thickness of walls, different ores used, date of receipt of the article, length of time the article is stored between sanding and enameling (danger of rusting), method of welding, any difference in models, cracks, cemented places, spots, rust, oil, irregularities, tensions and cracks occurring during cooling, hardness or brittleness of metal, porosity, etc. Only by careful control can many defects be eliminated and high quality products be obtained at a relatively moderate cost. M.V.C.

Review of current porcelain literature. M. E. BUTLER. *Jour. Can. Ceram. Soc.*, **9**, 56–62 (1940).—B. reviews the outstanding developments in porcelain enameling occurring during the previous year. J.G.P.

Solubility of zirconium oxide opacifier in enamels. V. *Emailwaren-Ind.*, **18** [3–4] 5–6 (1941).—The extent to which zirconium oxide dissolves in enamels is discussed mainly on the basis of the work of A. I. Andrews and R. W. Gates (*Jour. Amer. Ceram. Soc.*, **23** [10] 288–90 (1940)). M.V.C.

Use of nepheline-syenite in enamels for cast iron. HARRY C. PRIEST. *Jour. Can. Ceram. Soc.*, **9**, 53–55 (1940).—The results of a study to determine the effect of replacing feldspar with nepheline-syenite in ground-coat enamels for cast iron are as follows: (1) The group of ground coats containing 80% nepheline-syenite and 20% flint closely approximated the ground coat containing all feldspar; as flint is cheaper than feldspar a saving can be made without altering the firing time or temperature. (2) The straight replacement of feldspar with nepheline-syenite did not give as good results as reduced amounts of nepheline-syenite or a mixture of the latter with feldspar. (3) The best ground coats, with regard to low fusion and long vitrification range, were given by the groups having all the feldspar replaced by 90% and 80% nepheline-syenite and that having 75% of the feldspar replaced by nepheline-syenite. (4) All ground coats when fired to maturity had a high degree of adherence. (5) Although syenite A was superior to syenites B or D for ground coats, the difference was not great enough to warrant the use of a more expensive material. Syenite D, the cheapest of the samples used, gave excellent results. The more coarsely ground syenite B may be readily used in place of syenite A, its fusibility being only slightly less. J.G.P.

PATENTS

Apparatus for abrasively treating metal castings, forgings, etc. D. C. TURNBULL (American Foundry Equipment Co.). U. S. 2,240,248, April 29, 1941 (Feb. 17, 1938).

Apparatus for abrasively treating metal objects. C. H. HAMMELL (American Foundry Equipment Co.). U. S. 2,239,714, April 29, 1941 (Jan. 29, 1937).

Apparatus for cleaning metal articles. C. H. HAMMELL (American Foundry Equipment Co.). U. S. 2,240,246, April 29, 1941 (June 12, 1939).

Mottled enamelware. E. E. BRYANT (Ferro Enamel Corp.). U. S. 2,241,803, May 13, 1941 (Nov. 7, 1938).

Vitreous enamel. V. H. REMINGTON AND RAY ANDREWS (B. F. Drakenfeld & Co., Inc.). U. S. 2,241,520, May 13, 1941 (June 11, 1936).—A vitreous enamel containing approximately 63 lead oxide, 31 to 33 silica, between 3 and 4 boric oxide, and approximately $1^1/_2$% sodium oxide.

Vitreous enamel tank connection and method of construction. O. E. UECKER (A. O. Smith Corp.). U. S. 2,239,509, April 22, 1941 (June 24, 1938).

Glass

Bells made from glass. ANON. *Rundschau Deut. Tech.*, **20** [28] 2 (1940); *Umschau*, **45** [3] 42 (1941).—Originally, lead glass was used, but new substitutes have replaced lead completely and satisfactorily. The composition is not given. See "Signal . . . ," *Ceram. Abs.*, **19** [9] 212 (1940). J.M.N.

Can minium be replaced in the production of lead glasses? V. ČTYROKÝ AND M. FANDERLIK. *Glashütte*, **71** [5] 53–56 (1941).—The authors attempted to find a glass with a greatly reduced lead content but with its appearance, optical properties, and specific gravity as similar to those of lead crystal glass as possible. Other requirements of the glass were stability to light and capacity to take a polish with hydrofluoric acid. Previously published data showed that glasses containing 4 to 6% PbO would have to be considered because of their color and light stability. Two series of glasses were studied: (*1*) *Baryta glasses:* Glass with a BaO content of up to 15% was chosen; with glass containing 4 to 6% PbO, the required index of refraction and specific gravity were obtained. The results showed that glasses with a foundation composition of 65 SiO_2, 15 BaO, 8 K_2O, and 6% Na_2O, if they contain 2 to 0% CaO and 4 to 6% PbO, will fill the requirements for lead crystal glasses (minimal index of refraction 1.53, minimal specific weight 2.78, stability to light, and capacity to take a polish with acid in the usual concentration) with the exception of the relative dispersion ($1/\nu$) which could not be completely obtained in industrial production with substitutes at a cost within reach. (2) *Lead glasses:* Two half-lead glasses with 6% and 11.3% PbO and without barium oxide and a lead glass with 22% PbO were used for comparison. The half-lead glasses without BaO filled none of the requirements of lead crystal glasses, with the exception of the color stability, and could not be polished with hydrofluoric acid. The addition of 15% BaO was approved for glasses with 4 to 6% PbO because it brought the index of refraction and the specific weight up to the required values and so accelerated the hydrofluoric acid polishing process that it was completed sooner than in the case of lead crystal glasses with 20 to 22% PbO. The following conclusions were drawn from measurements of the baryta-lead glasses: (*a*) The addition of PbO to baryta-lime glass with a constant content of SiO_2, BaO, Na_2O, and K_2O, with the replacement of CaO, raises the index of refraction for sodium light (n_D) in the range of the glasses tested from 1.519 to 1.537. The replacement of 1% CaO by 1% PbO increases n_D on an average of about 0.003. (*b*) The replacement of CaO by lead oxide raises the specific weight of the glass from 2.645 to 2.875; the replacement of 1% CaO by 1% PbO increases the specific gravity of the glass on the average of about 0.038. (*c*) The replacement of CaO by lead oxide raises the reciprocal relative dispersion ($1/\nu$). This increase is relatively small for small amounts of lead but becomes greater with larger amounts of PbO. The total increase for 0 to 6% PbO amounts to 0.0068. The increase of the reciprocal relative dispersion for 1% PbO amounts to 0.0005 within the limits 0 to 2% PbO, to 0.0014 for 2 to 4% PbO, and to 0.0016 for 4 to 6% PbO. (*d*) Beginning with 4% PbO the glasses are stable to light and are colorfast. The authors conclude that there is no substitute raw material, equivalent or accessible in pure form, for industrial production which can completely give the optical properties of lead crystal glasses made with minium; disregarding the property of the relative dispersion (required, 0.020; attainable, 0.013), however, lead crystal glasses can be successfully replaced by glasses containing 4 to 6% PbO and 15% BaO. M.V.C.

Chemical methods for increasing the transparency of glass surfaces. F. L. JONES AND H. J. HOMER. *Jour. Optical Soc. Amer.*, **31**, 34–37 (1941).—The amount of light transmitted by a lens or prism can be increased by treating the polished glass surface by a chemical process which involves the selective solution of some of the elements present in the glass, leaving a silica film. The process is analogous to weathering, but surface film thickness is controlled. When the greatest gain in transmission is required, evaporated films of minerals with low refractive index are preferable; chemical treatment films, however, are superior for hardness and durability. Suitable solvents include acid solutions, salt solutions, water, alkaline phosphate solutions, and fused salts. Dilute acid is best for lead, barium, or borosilicate glasses. Effects of solution and glass compositions and time and temperature of treatment are examined. The condition of the surface film before the start of the treatment has an important effect on the rate of reaction. When the acid treatment is followed by a firing operation, the silica film becomes so stable that no ordinary weathering will increase its thickness. A.P.

Colloidal mixtures as batches for glassmelting. F. DE KÖRÖSY. *Bull. Amer. Ceram. Soc.*, **20** [5] 162–63 (1941).

Corning's top-of-stove ware. ANON. *Glass Ind.*, **22** [4] 171–72 (1941).—Because it is subjected to localized heating, glass for top-of-stove ware requires special characteristics. Since it must hold its temper strain to at least 500°C., the strain temperature should be as high as 675°C. Viscosity should be high at the strain temperature. Thermal expansion coefficients should not exceed 65×10^{-7}. Suitable glasses and their properties are tabulated as follows:

TABLE I

	A	B	C	D
SiO_2	56.4	60.5	62.5	57.1
B_2O_3	5.0		5.2	12.4
Al_2O_3	23.0	21.4	15.6	15.5
Na_2O	0.8	0.6		
CaO	4.1	8.7	10.0	9.0
MgO	10.7	5.8	5.0	5.0
Fluorine		1.5	1.5	1.0

TABLE II

	A	B	C	D
Softening temp.	929	938	888	871
Annealing temp.	726	715	663	639
Strain temp.	684	672	625	603
Expansion ($\times 10^{-7}$)	38	41	40	39

Strain release is also governed by the length of the setting range between the softening and strain temperatures. This should not exceed 300°. A graph shows the percentage of stress remaining after 100 hr. at 250° to 500° for glasses A to D and for four others with lower strain temperatures. The method of manufacture, as applied to glass A, is briefly described. A.P.

Electric breakdown of glass with impulse voltage. K. INADA AND T. SOIJIMA. *Electrotech. Jour.* [Japan], **4** [10] 231 (1940).—Experiments on window glass about 5 mm. in thickness were carried out with an impulse voltage of different wave form and an a.-c. voltage of 50 cycles. The samples were inserted between plate and needle electrodes immersed in transformer oil. With an impulse voltage of very steep wave (about 10^{-8} sec.), a partial breakdown could be developed at a comparatively low voltage (17 kv.) and the breakdown was perfect at about 80 kv. With a flat wave (about 10^{-6} sec.), surface corona appeared at about 45 kv., but the streamer did not penetrate into the glass at this voltage; the breakdown became suddenly complete at about 50 kv.; it was difficult to produce a partial breakdown. With an a.-c. voltage also, the streamer did not penetrate into the glass; the breakdown became complete between 50 and 60 kv. W.J.L.

Glass for artificial eyes. R. J. MONTGOMERY AND CLIFFORD TAYLOR. *Jour. Can. Ceram. Soc.*, **9**, 46–50 (1940).—Most of the glasses needed for the production of artificial eyes are of an unusual type, not ordinarily made commercially. The principal glass, produced in tube form and used for "bulbs," is opalescent, and the density of the opalescence must be under control so that the worker may obtain, by proper heat treatment, a range from clear glass to dense opal at will. The production of such glass has been confined to a few plants in Europe. Analyses of several of the foreign glasses and numerous experimental melts were made; this led to the development of a glass suitable for the purpose in all respects. A plant in the U. S. has consented to produce the glass commercially, bridging the gap between laboratory experimental work and factory production by further experimentation if required. J.G.P.

Heat-transfer coefficients in glass exchangers. A. S. FOUST AND T. J. THOMPSON. *Trans. Amer. Inst. Chem. Engrs.*, **36**, 555–75 (1940).—The results of heat-transfer measurements in double-pipe exchangers, the inner pipes being Pyrex brand tubes 1 in. and 2 in. in diameter, are given. Evidence indicating that the thermal conductivity value of Pyrex brand borosilicate glassware is higher than published values is discussed. H.E.S.

History of glassworking. HANS SCHULZ. *Glastech. Ber.*, **19** [1] 8–9 (1941). J.F.H.

Humidity effect on the loss angle of insulators at high frequencies. T. MIURA AND T. KAMBARA. *Electrotech. Jour.* [Japan], **3** [10] 238–39 (1939).—The loss angle of borosilicate glass was determined at 300 to 10,000 kc. and 30 and 95% relative humidity. The humidity effect was marked at low frequencies but became very small with increasing frequencies. As the material does not absorb moisture, the variation of the loss angle may be regarded as being due to surface leakage. The effect calculated under this assumption is in good agreement with experimental data. W.J.L.

Light globes of flash glass and clear glass. HEINRICH FUCHS. *Glashütte*, **71** [4] 42–43 (1941).—Light globes of flash glass and large outer globes of clear glass ornamented with thick horizontal bands of cream glass or thin bands of frosted glass are illustrated. M.V.C.

Manhasset modernizes. ANON. *Amer. Builder*, **61** [8] 64, 65 (1939).—Old stone fronts were replaced with ivory and black Vitrolite structural glass. Replacement was made without loss of time to tenants. See "Vitrolite . . . ," *Ceram. Abs.*, **20** [3] 66 (1941). S.S.

Matting and decorating glass by sandblasting. R. K. *Glashütte*, **71** [3] 30 (1941).—Vacuum sandblasting is used more often than compressed air or steam sandblasting for finishing flat glass. The operation of the apparatus is as follows: The sheet of glass is rested on rubber rollers over the nozzle, and a vacuum is created in the space beneath the glass with a vacuum pump; this produces a powerful air current which carries the sand through the nozzle against the sheet of glass. Special attachments for the nozzle produce a narrow or broad jet to make fine or broad lines on the glass. By concentrating the blast on a small area, a hole 2 to 50 mm. in diameter can be bored through the glass in 1 or 2 min.; compressed air or steam sandblasting is usually used for this purpose. When an ornamental pattern is to be made on the glass, stencils of metal are placed over the glass before blasting, or a protective paint is applied to the parts of the glass surface which are not to be blasted. The paint should be tough but not hard or brittle. Two recipes for protective paint are given: (1) 50 gm. gelatin, 50 cc. 25 degree zinc chloride solution, 50 cc. 25 degree glycerin, and 250 gm. Swedish pine soot; (2) 500 gm. fine clay mixed with 250 cc. water and heated; 50 gm. glue and 100 cc. glycerin are added and mixed well; the mixture is heated until enough water has evaporated to give the proper consistency. This produces an excellent resistant mass. After sandblasting, the paint is removed by washing with water. Mat decoration or inscription may be gilded, bronzed, or painted with silver and other colors. All kinds of glass signs, advertisements, etc., may be finished in this way. M.V.C.

Method of evaluating the characteristics of ground glass surfaces. S. F. LEGUN. *Optiko Mekh. Prom.*, **8** [6] 1–2 (1938); abstracted in *Physik. Ber.*, **20** [13] 1364 (1939).—The grinding process is judged by (1) the amount of glass ground away in unit time and (2) the quality of the surface left. M.H.

Methods and compounds for glass frosting or etching. J. F. MAGURNO. *Amer. Glass Rev.*, **60** [26] 19–20; [27] 20–21 (1941).—Because of the special equipment and the hazards involved in the use of formulas containing hydrofluoric and sulfuric acids, solutions based upon ammonium bifluoride are now being used. The addition of a filler to check fuming further decreases hazards and prolongs the effectiveness. A.P.

Miami's wonder house. ANON. *Amer. Builder*, **62** [7] 43–45 (1940).—Small homes recently built in Miami, Florida, contain two and a quarter times as much glass as the average $5500 house. Mirrors, large windows, and glass doors make the interior appear larger. S.S.

Millville's new furnace room. ANON. *Glass Ind.*, **22** [4] 151–53 (1941).—The Armstrong Cork Co. has consolidated its two Whitall-Tatum plants in Millville, N. J. A new furnace room has been built as the first step in a three-year modernization program. Eight photographs. A.P.

Organization of research departments. F. W. PRESTON. *Glass Ind.*, **22** [4] 163–65, 175 (1941).—Signs point to the necessity of the glass industry to spend more effort and money on scientific research. P. considers some of the problems and procedures involved, using as a model a container company doing a $10,000,000 annual business. A.P.

Platinized glass as a laboratory substitute for massive platinum. C. C. COFFIN. *Can. Jour. Research*, **18B**, 318–21 (1940); *Chem. Abs.*, **35** [2] 348 (1941).—A method for sealing Pt through Pyrex brand glass and the use of platinized glass in the construction of various laboratory devices are described.

Report of the International Commission of Glass Technology. J. C. HOSTETTER, *Chairman*. *Bull. Amer. Ceram. Soc.*, **20** [5] 176–77 (1941).

Role of zinc oxide in sulfide or selenide glasses. Reactions on warming-in selenium glasses. A. DIETZEL. *Glastech. Ber.*, **19** [1] 1–4 (1941).—The presence of ZnO in sulfide or selenide glasses serves the special purpose of preventing the burning out of S or Se and suppresses the color in the so-called carbon glasses. This action is satisfactorily explained by the formation of sulfozincate and selenozincate complexes of the type $(ZnS_4)^{6-}$. The selenium complexes are more stable than the sulfur complexes. ZnS can thus separate out of the melt, especially in more acid glasses. In selenium rubies, the cadmium behaves similarly, but its sulfur and selenium complexes are less stable than those of Zn. J.F.H.

Sulfide, selenide, and telluride colors in glasses. A. DIETZEL. *Glastech. Ber.*, **19** [1] 4–8 (1941).—The solubility of heavy metal sulfides, selenides, and tellurides in glass is related to their ability to form stable coordination complexes of the type $(MeX_4)^{6-}$. Such complex formation appears to be possible when the ratios of the ionic radii Me^+/X^- are between 0.225 and 0.414; this is a factor in determining their relative stabilities and thus their solubilities in glass. The stabilities of the complexes increase with increasing basicity of the base glass. J.F.H.

Survey of the health situation in the glass industry. R. PLUHAR. *Glastech. Ber.*, **19** [1] 10–15 (1941). J.F.H.

Vanadium and chromium oxides in refractories as aids in the elimination of cords in glass. A. E. BADGER AND H. R. PINNOW. *Glass Ind.*, **22** [4] 161–62 (1941).—The incorporation in refractories of oxides which produce on solution a lower surface tension in the glass is suggested as a means of hastening the solution of cords caused by the corrosion of refractories. Vanadium oxide and especially chromium oxide were suitable. Glasses melted in pots containing chromium oxide were colorless except for a slight fringe of green at the metal line. A.P.

BOOKS

Key to Pottery and Glass. BERNARD RACKHAM. Chemical Publishing Co., Inc., New York, 1941. 180 pp., 17 illustrations. Price $2.25. See *Ceram. Abs.*, **19** [10] 229 (1940).

Modern Glass Practice. S. R. SCHOLES. Industrial Publications, Inc., Chicago, 1941. Revised ed. 289 pp. Price $6.00.—Each chapter has been expanded, corrected, and brought completely up to date in the light of recent developments. The number of illustrations has been increased, and many of the old ones have been replaced with better ones. New information on raw materials, convection currents, batch compositions, applications of glass, and absorption in colored glasses has been added. A better grade of paper and a more attractive binding are also used in this excellent book. For review of first edition see *Ceram. Abs.*, **15** [4] 120 (1936).

PATENTS

Alloy for metal-to-glass seals. V. O. ALLEN AND C. P. MARSDEN, JR. (Wilbur B. Driver Co.). U. S. 2,240,063, April 29, 1941 (Sept. 6, 1940). U. S. 2,240,064, April 29, 1941 (Sept. 6, 1940; Feb. 6, 1941).

Apparatus for handling glassware. O. M. FARRIS (Hazel-Atlas Glass Co.). U. S. 2,240,093, April 29, 1941 (Aug. 30, 1939).

Apparatus for making sliver. G. M. LANNAN AND FRANK VANUCCI (Owens-Corning Fiberglas Corp.). U. S. 2,239,722, April 29, 1941 (May 21, 1937; renewed Nov. 13, 1939).

Apparatus for making tempered glass. G. Z. MINTON AND L. V. BLACK (Pittsburgh Plate Glass Co.). U. S. 2,239,535, April 22, 1941 (March 25, 1938).

Apparatus for shaping glass charges. J. W. ROSS (Hazel-Atlas Glass Co.). U. S. 2,241,162, May 6, 1941 (March 23, 1938).

Cement for glass. I. D. BUTYRKIN. Russ. 55,150, June 30, 1939; *Chem. Abs.*, **35**, 3008 (1941).—An adhesive, especially for optical glass, consists of fir balsam treated with linseed oil and boiled.

Electric glassmelting furnace. A. R. MELIK-AGAMIRYAN. Russ. 54,714, April 30, 1939; *Chem. Abs.*, **35**, 3052 (1941).

Glass. RUDOLF SCHMIDT AND WALTER HÄNLEIN (General Electric Co.). U. S. 2,240,352, April 29, 1941 (Feb. 8, 1938).—A glass resistant to hot ionized alkali metal vapor when at a temperature of 300°C., the glass being capable of fusion with a nonalkali vapor-resistant soft soda glass having a coefficient of expansion of about 80 to 90 $\times 10^{-7}$ to form a two-layer glass container for an alkali vapor discharge lamp, the resistant glass consisting of about 20 to 30 boric anhydride (B_2O_3), about 20 to 30 alumina (Al_2O_3), about 4 to 9 silica (SiO_2), about 30 to 40 alkaline earths, and about 6 to 10% alkalis.

Glass article and process for manufacturing. COMPAGNIE DES VERRERIES DE MOUTIER POUR LA FABRICATION MÉCANIQUE DU VERRE (Procédés Libbey-Owens) Soc. ANON. Brit. 535,155, April 9, 1941 (Feb. 8, 1939).

Glass-block mounting. WILLIAM OWEN (Pittsburgh Plate Glass Co.). U. S. 2,239,537, April 22, 1941 (April 1, 1939).

Glass-block structures and process and glass blocks for making. CORNING GLASS WORKS. Brit. 535,207, April 17, 1941 (April 18, 1939).

Glass composition. WILLEM ELENBAAS AND G. B. JONAS. U. S. 2,240,327, April 29, 1941 (July 28, 1936; April 16, 1938).—A glass contains silica (SiO_2) 78, alumina (Al_2O_3) 12, and calcium oxide (CaO) 10%.

Glassforming machine. S. D. BERT (M. A. Yorkin and J. D. Martin). U. S. 2,240,324, April 29, 1941 (April 21, 1939).

Glass furnace provided with drawing-off basins. EMILE ROIRANT (Société Continentale d'Appareils Mécaniques pour la Verrerie). U. S. 2,241,161, May 6, 1941 (Jan. 6, 1937).

Glassmelting furnace. P. G. WILLETTS (Hartford-Empire Co.). U. S. 2,240,361, April 29, 1941 (April 26, (1938).

Glassmelting furnace with water cooling. S. I. KOROLEV. Russ. 54,761, April 30, 1939; *Chem. Abs.*, **35**, 3052 (1941).

Iron-glass seal having nickel-iron contact prongs. A. W. HULL (General Electric Co.). U. S. 2,239,423, April 22, 1941 (April 7, 1939).

Laminated glass. ADOLF WEIHE AND FRITZ HERRLEIN (General Aniline & Film Corp.). U. S. 2,242,019, May 13, 1941 (April 11, 1939).

Machinery for the manufacture of glass wool. D. P. ORLOV AND A. S. PRYANISHNIKOV. Russ. 54,712, April 30, 1939; *Chem. Abs.*, **35**, 3053 (1941).—Constructional details are given.

Making sealing glasses and seals for quartz lamps. R. H. DALTON AND M. E. NORDBERG (Corning Glass Works). U. S. 2,239,551, April 22, 1941 (April 22, 1939).

Means for making mineral wool. C. F. RAMSEYER. U. S. 2,242,089, May 13, 1941 (Aug. 13, 1938).

Method and apparatus for burning off glassware. H. R. SCHUTZ (Libbey Glass Co.). U. S. 2,239,627, April 22, 1941 (Sept. 30, 1938).

Method and apparatus for firing and feeding batch to glassmelting furnaces. VERGIL MULHOLLAND (Hartford-Empire Co.). U. S. 2,241,611, May 13, 1941 (June 9, 1938).

Method and apparatus for making optical glass. J. C. HOSTETTER (Corning Glass Works). U. S. 2,242,129, May 13, 1941 (Feb. 7, 1939).

Method and apparatus for making structural glass. F. W. PRESTON. U. S. 2,240,349, April 29, 1941 (Oct. 14, 1938).

Mirrors. N. A. BELOZERSKII. Russ. 54,429, Jan. 31, 1939; *Chem. Abs.*, **35**, 3052 (1941).—A layer of Ag is precipitated on glass by chemical means, and a second layer is then precipitated galvanically on the first layer to permit polishing. A Cu layer is finally placed galvanically on the second Ag layer.

Multiple laminated glass. J. W. KAMERER (Pittsburgh Plate Glass Co.). U. S. 2,239,527, April 22, 1941 (July 12, 1938).

Optical glass. L. W. EBERLIN AND P. F. DE PAOLIS (Eastman Kodak Co.). U. S. 2,241,249, May 6, 1941 (July 12, 1939).—A glass having an index of refraction for the D line of about 1.72 and a dispersive index of about 47 and resulting fusion of a batch containing between 15 and 35 lanthanum oxide, between 5 and 15 thorium oxide, between 28 and 37 boric oxide, between 10 and 20 of oxides from the group consisting of tantalum oxide and tungsten oxide, and between 5 and 25% of oxides from the group consisting of barium, calcium, strontium, and magnesium oxides.

Optical glass. KODAK SOC. ANON. Swiss 206,664, Dec. 16, 1939; 11*b*; *Chem. Abs.*, **35**, 3052 (1941).—The glass contains at least 10% SiO_2 and at least one oxide of a metal belonging in the first subgroups of groups III to VI of the periodic system, the metal having an atomic weight of over 47. The refractive index of the glass is at least 1.68, and the dispersion exponent is $[4.6/(n_D-1.6)] + 7$. Examples are given of batches containing Ti or Ta and Ti.

Process and apparatus for bending glass sheets. L. V. BLACK AND G. Z. MINTON (Pittsburgh Plate Glass Co.). U. S. 2,239,546, April 22, 1941 (March 30, 1938).

Tempering glass tubing. KENNETH STRATTON (Corning Glass Works). U. S. 2,239,796, April 29, 1941 (March 22, 1938).

Treating the surface of glass bodies. J. F. GREENE (Kimble Glass Co.). U. S. 2,241,511, May 13, 1941 (Sept. 29, 1938).

Vertical furnace for annealing glass, etc. A. A. ZALIZNYAK. Russ. 55,078, June 30, 1939; *Chem. Abs.*, **35**, 3052 (1941).

Water- and steam-resistant glass. GLASFASERN A.-G. Swiss 207,325, Feb. 1, 1940; 11*b*; *Chem. Abs.*, **35**, 3052 (1941).—A glass batch contains acids that lower the viscosity of the molten glass, together with heavy metal oxides and alkali oxides. An example is Hohenbockaer sand 373, feldspar sand 210, calcite 32, potash 14, $NaNO_3$ 17, Na_2CO_3 122, B_2O_3 122, and ZnO 105 parts by weight. Glass from this batch is easily spun or made into glass wool.

Structural Clay Products

Cedar Rapids, Iowa, improves principal downtown street with paving brick. ANON. *Dependable Highways*, No. 170, pp. 3, 15 (1941). P.S.D.

PATENTS

Building brick. W. H. WHITTEN. U. S. 2,241,079, May 6, 1941 (May 11, 1939).

Drainage tile, clay, etc. L. L. NEWMAN. U. S. 2,240,411, April 29, 1941 (June 12, 1940).

Hollow brick building block and reinforced brick wall. J. B. PEEBLES (Gladding, McBean & Co.). U. S. 2,239,930, April 29, 1941 (Dec. 5, 1939).

Refractories

Basic brick vs. design in checker-brick efficiency. C. E. GRIGSBY. *Glass Ind.*, **22** [4] 157–60 (1941).—In the glass industry, a superior refractory or a special shape and method of setting which would increase the heating surface while making it easier to clean out the carry-over were sought in attempts to increase the efficiency of the regenerators. Nonbasic refractories are sticky enough at top checker temperatures to trap dry carry-over. With basic brick, the batch dust passes out of the checker chamber, and the brick remain full size, clean, and efficient throughout the run. Basic brick also have a heat capacity 30 to 50% greater than that of fire-clay brick. Fired basic refractories have low resistance to thermal shock, but chemical bonding of the brick has overcome this defect. Plant tests which have been under way since 1933 have led to special mixtures for this specific purpose. A.P.

Carbon linings for blast furnaces. F. J. VOSBURGH. *Iron & Steel Engr.*, **17**, 68–72 (April, 1940).—Since 1890, three types of carbon linings have been tried: (1) standard size brick; (2) carbon blocks up to 3000 lb., accurately machined and laid with a minimum of cement; and (3) a carbon paste tamped into place. The second type is preferred. Ceramic tap holes are used. The furnace is protected during blowing-in by a wall of 5-in. firebrick. All cooling is done by a spray or shower on the outside of the shell. The cost of a furnace producing 1000 tons per day is estimated at $75,000. The probable life is estimated at 2 to 3 million tons with few, if any, repairs. The advantages include no loss of metal, no hot spots, no cooling plates, and no complicated piping to maintain. B.L.

Corundum brick, a highly refractory material for the lining of rotary cement kilns. M. A. FRAĬFELD. *Prom. Stroitel. Materialov*, **2** [9] 5–9 (1940).—Corundum brick prepared from ground electrically fused corundum bound with refractory clay is highly refractory, is resistant to spalling and clinker attack, and has a high mechanical strength. It is believed to be very resistant in rotary cement kiln linings. V. D. SIDOROV. *Ibid.*, pp. 9–10. P.B. & E.S.

Decreasing the flaws in glass by the use of new refractories. A. A. LITVAKOVSKII. *Prom. Stroitel. Materialov*, **2** [9] 49–53 (1940).—L. recommends the use of vacuum-cast blocks, coarse-grained rammed blocks, electrically fused corundum blocks, rammed crucibles, acid clays for the production of crucibles, new masses for the production of Fourcault boats, electrically dried crucibles, and silicon carbide refractories for saggers. P.B. & E.S.

Developments in the refractories industry. ANON. *Refrac. Jour.*, **17** [2] 47 (1941).—Developments in the manufacture of refractories in the U. S. in 1939 to 1940 are discussed. B.C.R.

Durability of bottoms of acid open-hearth furnaces. V. MAKSIMOV. *Stal*, **1939**, No. 4–5, pp. 18–19.—M. studied the influence of sand with varying SiO_2 content on the durability of bottoms of acid open-hearth furnaces. The durability decreases with increasing SiO_2 content (90 to 95%). M. recommends the addition to the sand of 5 to 10% ground refractory clay of the following composition: SiO_2 50 to 52.5, Al_2O_3 31 to 32.5, Fe_2O_3 0.5 to 0.7, and CaO + MgO 3%. B.E.K.

Extraction of alumina from kaolin and other silicates. K. KAMMERMEYER AND A. H. WHITE. *Trans. Amer. Inst. Chem. Engrs.*, **36**, 683–99 (1940).—Al_2O_3 can be extracted from kaolin, coal ash, and similar aluminum silicates by firing a mixture of aluminous material, limestone, and soda ash in a rotary kiln and subsequently leaching the resulting clinker. The Al_2O_3 recovered will contain no more SiO_2 and CaO than that now used as a raw material in the aluminum industry. Extractions of 85 to 90% of the total Al_2O_3 and Na_2O in the clinker are possible if the composition of the mix and the firing conditions are properly adjusted. Wide variations from the molecular ratios of $2CaO:1SiO_2$ and $1Na_2O:1Al_2O_3$, as well as high percentages of Fe_2O_3, are likely to give low yields of Al_2O_3. A temperature higher than that which causes sticking of the clinker in the rotary kiln may be beneficial to the recovery of Al_2O_3. It is probable that fusion in an electric furnace would give better recoveries than those obtained by firing in a rotary kiln. Commercial possibilities of the process are dependent on the full utilization of waste products. The use of the insoluble residue in the manufacture of Portland cement appears to offer the greatest possibilities of successful operation. H.E.S.

Increase in the solubility of the alumina in kaolins as a result of preheating in the presence of gaseous catalyzers. GUSTAV F. HÜTTIG AND ERICH HERRMANN. *Ber. Deut. Keram. Ges.*, **21** [11] 429–41 (1940).—Kaolin as it occurs in nature is not appreciably soluble even in strong acids. The alumina content becomes soluble when the kaolin is heated to about 500°C. The effect of calcining the kaolin in an atmosphere of air and of other gases, viz., N_2, O_2, NO_2, $(4NO_2 + O_2)$, SO_2, SO_3, H_2O, HCl, and NH_3, was studied. Four kaolins were used. The hydration took place in three steps: (1) about $^1/_8$ the entire amount of water was driven off below 300°C., (2) the bulk of the water (about $^6/_8$) was driven off between 400° and 600°C., and (3) the remaining water was removed very slowly as the temperature increased. The rate of formation of the alumina soluble in HCl depends largely on the catalyzer. The formation of soluble silica by calcining depends upon the gas used as a catalyzer. The period of heating has a marked effect, e.g., long heating at relatively low temperatures produces preparations containing more soluble alumina than those produced by shorter calcining at higher temperatures. For a given preparation, the alumina soluble in H_2SO_4 is almost always nearly equal to the alumina soluble in HCl. The sorption capacity, at least for preparations heated to not over 600°C., depends largely on the catalyzing gas. The maximum sorption capacity was found for preparations heated to 300°C. Obviously, the first step in dehydration is a process connected with a marked activation followed by a deactivation. It is concluded that the physical and chemical characteristics of the dehydrated product can be influenced by the gaseous atmosphere in which kaolin is calcined. W.H.H.

Investigation of the forsterite (dunite) lining of a rotary kiln at the Kramatorsk Works. V. S. GULYAEV, V. YA. MEFODOVSKIĬ, AND I. K. MOROZ. *Prom. Stroitel. Materialov*, **2** [9] 10–12 (1940).—The results obtained, though not definitely conclusive, show the high quality of forsterite refractories for rotary cement kiln linings. P.B. & E.S.

Life and disadvantages of refractories in the metallurgy of special steels. A. ŽHAK. *Stal*, **1939**, No. 4–5, pp. 50–60.—Various refractories, their application in metallurgical aggregates, and their disadvantages are discussed. The following refractories are reviewed: (1) silica brick, (2) siphon brick, (3) stoppers, (4) grog brick, (5) andalusite stoppers, (6) carbon grog brick, (7) magnesite brick, (8) dolomite brick, and (9) dunite brick. See "Quality . . ." *Ceram. Abs.*, **20** [5] 118 (1941). B.E.K.

Linings for the sintering zone of rotary kilns in the cement industry. P. P. BUDNIKOV AND V. I. ENDOVITSKIĬ. *Repts. (Dopovidi) Acad. Sci. Ukrainian S.S.R.*, **1940**, No. 4, pp. 35–40 (in Russian 40–41; in German 42–46).—To determine the most appropriate lining materials, the following materials were tested: dolomite brick, grog kaolin of increased density with 41.5% Al_2O_3, high-alumina brick containing 56.73% Al_2O_3, and forsterite brick. The latter was prepared from a batch of Ural dunite fired at 1450° with additions of quartzite, caustic magnesite, and molasses and mixed with a $MgCl_2$ solution. The brick was fired at 1600°. The properties were as follows: refractoriness P.C.E. 37 to 38, mechanical strength 160 to 300 kgm. per cm.^{2}, deformation under load beginning at 1620° to 1660° and ending at 1720° to 1770°, volume weight 2.45, and volume porosity 25.28%. The brick consists of forsterite, a small amount of fayalite, and periclase. It reacts slightly with Portland cement clinker at 1500°. P.B. & E.S.

Methods for the qualitative evaluation of silicates as raw materials for the production of Dinas stone. V. A. BRON. *Sovet. Geol.*, **9** [10–11] 121–24 (1939); *Chem. Abs.*, **35** [2] 591 (1941).—The method of the Ukrainian Institute for Scientific Research of Refractory and Acid-Resisting Materials consists of determinations of surface impurities, macro- and micro-structure, specific gravity, volume weight and porosity before and after firing, adsorptive capacity for H_2O, and determination of Al_2O_3 and Fe_2O_3 in all cases and of CaO and TiO_2 in certain cases.

Refractories industry of Canada. JAMES W. CRAIG. *Jour. Can. Ceram. Soc.*, **9**, 21–27 (1940).—The refractories industry in Canada is an essential key industry. The average annual consumption of refractories in Canada is about $3,000,000, compared with $100,000,000 for the U. S. In analyzing the refractories industry of Canada, C. presents significant statistics and comments. Canada, in a normal peace-time year, consumes about $1,500,000 worth of fire-clay brick, $350,000 worth of silica brick, and $1,000,000 worth of basic refractories. In 1939, Canada produced about $600,000 worth of fire-clay brick, $125,000 worth of silica brick, and $675,000 worth of basic refractories. Canada is exporting annually between $200,000 and $250,000 worth of basic refractories. The ratio of basic refractories to fire-clay and silica brick used in Canada is much higher than that in the U. S., Great Britain, France, and Germany. This is due to the relative size of the metallurgical industry compared with other large refractory-consuming industries and to the use of hydroelectric power rather than refractory-consuming steam-generated power. In recent years there has been a steady and marked replacement of silica brick by basic refractories in Canadian steel furnaces; this trend will probably continue. Canada suffers from lack of high-grade and geographically well-placed fire-clay deposits. J.G.P.

Refractory cement and concrete. S. N. DUGUID. *Steam Engr.*, **9**, 402–404 (1940).—D. lists the properties of refractory concrete made from aluminous cement and aggregate chosen from fire-clay grog, ground fire-clay brick, sillimanite, chrome, and chrome magnesite. One cement had the following analysis: SiO_2 5.55, Al_2O_3 36.67, Fe_2O_3 11.60, FeO 5.13, TiO_2 2.08, and CaO 38.75%. The melting point is above 1450°C. A concrete mix suitable for most purposes contains coarse aggregate ($^3/_4$ to $^1/_8$ in.), 3 cu. ft., fine aggregate ($^1/_8$ in. down), 2 cu. ft., and refractory cement, 1 cu. ft. This concrete has a bulk density of 90 lb. per cu. ft. A mortar contains 1 cu. ft. of cement to $2^1/_2$ to 4 cu. ft. of grog ($^1/_8$ in. down). The cement may be used for laying up firebrick or for hot patching. The concrete can be used for foundations, walls, arches, and doors of furnaces and for flue linings. See also *Ceram. Abs.*, **20** [5] 118 (1941). L.R.B.

Spalling resistance of fused silica vs. quartz and calcined fire-clay grog. W. A. SEMPLE AND R. J. MONTGOMERY. *Jour. Can. Ceram. Soc.*, **9**, 40–42 (1940).—The comparative effect of the use of fire-clay grog, quartz, and fused silica on the properties of an assay crucible body was studied. In all cases the grog was graded to minus 20-mesh, plus 65-mesh. The results indicate that there is little chemical reaction between silica, introduced either as quartz or fused silica, and the bond clay. The resulting body is porous, with grog loosely held in the plastic material. The introduction of either form of silica increases the resistance to spalling, the fused silica being much more effective than crystalline quartz. J.G.P.

Specific heats of chrome and magnesia refractories. GILBERT E. SEIL, FRANK G. HECK, AND H. A. HEILIGMAN. *Jour. Amer. Ceram. Soc.*, **24** [6] 204–12 (1941).—8 figures, 128 references.

Steel-plant refractories. J. H. CHESTERS. *Iron Age*, **147** [6] 33–36; [7] 47–52 (1941).—The variability of refractories is an important factor in the production and quality of steel. The following tests are useful: apparent porosity, bulk density, true specific gravity, cold crushing strength, permeability to air, after shrinkage or expansion, melting point of pyrometric cone, refractoriness under load (rising temperature), refractoriness under load (maintained temperature), thermal-shock resistance, slag resistance, microscopical examination, and X-ray examination. Each test is discussed briefly. References are given. E.H.McC.

Testing of new lining materials at the Kramatorsk Cement Works. A. S. SOKOLOV. *Prom. Stroitel. Materialov*, **2** [9] 13–16 (1940).—Linings of unfired chromite, unfired chromite-magnesite, and dunite brick proved to be more resistant than those of chromite refractories with alumina cement binding; the latter, however, are more resistant than the usual clinker cement linings. P.B. & E.S.

SEPARATE PUBLICATION

Superduty Fire-Clay Brick. S. M. SWAIN. *Amer. Refrac. Inst. Tech. Bull.*, No. 76, 7 pp. (Feb., 1941).—Superduty fire-clay brick may be considered as a superior fire-clay brick, as a brick which retains its outstanding resistance to spalling, slagging, and volume change at higher temperatures than first quality brick, or as a brick made of superior raw materials under extremely careful control. The definition depends largely on whether the viewpoint is that of the user or of the operator. The American Society for Testing Materials has classified it as "fireclay brick having a pyrometric cone equivalent not lower than cone No. 33 on the fired product, not more than 1% linear shrinkage in the permanent linear change test, schedule C (1600°C., 2912°F.), and not more than 4% loss in the panel spalling test (preheated at 1650°C., 3002°F.)." Practically all of the superduty brick are made by the dry-press process because of the ability of that process to form firebrick from coarse-grained clay bodies. The details of manufacture are described. The outstanding properties of superduty fire-clay brick are its high resistance to spalling and slagging and its ability to bear extreme loads at high temperatures. H.E.S.

PATENTS

Apparatus for casting refractories. R. C. BENNER AND J. C. MCMULLEN (Carborundum Co.). U. S. 2,241,386, May 13, 1941 (Nov. 9, 1937; March 28, 1938).

Ceramic molded body having high heat-conductance properties. STEINZEUGFABRIK EMBRACH A.-G. FÜR KANALISATION UND CHEMISCHE INDUSTRIE. Swiss 206,140, Jan. 2, 1940; *8b; Chem. Abs.*, **35**, 3054 (1941).—Si or its alloys are calcined in an atmosphere containing O at such a temperature that a minimum porosity without deformation and cracking of the calcined material is assured. In this way the calcination speed is so selected that the oxidation of the Si or of the Si alloys reaches only an intermediate stage which does not effect an impairment of the heat conductance and does not produce an inner tension in the material. The porosity of the calcined product can be further lessened by impregnating the calcined molded body with an organic liquid rich in hydrocarbons and heating it up to complete coking.

Furnace structure. L. S. LONGENECKER. U. S. 2,240,190, April 29, 1941 (Dec. 5, 1939).—In a furnace structure, a support beam, a furnace roof portion supported from the beam, a hanger supported from the beam, a vertical wall supported from the hanger, a series of pairs of refractory blocks forming the furnace nose and closing the space between adjacent ends of the furnace roof and the vertical wall, each such pair of refractory blocks comprising an upper block primarily supported by the hanger and a lower block supported by the end block of the furnace roof.

Furnace wall. M. H. KUHNER AND G. A. REHM (Riley Stoker Corp.). U. S. 2,239,896, April 29, 1941 (July 4, 1939).

Furnace wall or similar structure. B. L. CASSADY. U. S. 2,239,588, April 22, 1941 (April 17, 1939).

Making high-temperature refractories. R. E. LOWE AND A. G. GRISWOLD (Cities Service Oil Co.). U. S. 2,241,354, May 6, 1941 (Feb. 8, 1938).—A method of making a high temperature resistant refractory article comprises heating a metal containing substantial amounts of silicon in finely divided state in the presence of oxygen for several hours at a temperature above 2000°F. at which partial oxidation takes place forming oxide-coated particles, mixing about 75 to 85 parts by weight of the oxide-coated metal particles with about 9 to 19 parts of finely ground zircon and 2 to 4 parts each of colloidal clay and glass, wetting the mixture to a plastic state, shaping the article, and firing the article to a temperature sufficient to intimately bond the ingredients thereof.

Manufacturing insulating firebrick. C. L. NORTON, JR. (Babcock & Wilcox Co.). U. S. 2,242,434, May 20, 1941 (Feb. 27, 1937).—A method of making a ceramic product comprises intimately dry-mixing plaster of Paris, wood flour, and Georgia kaolin in the ratio of 37 lb. of plaster of Paris, 37 lb. of wood flour, and 100 lb. of Georgia kaolin, all in pulverized form, adding 100% water while mixing to form a flowable castable mass, molding shapes therefrom prior to recrystallization of the plaster of Paris, firing the shapes at a firing temperature in a reducing atmosphere while the mixture still retains substantially all of its water and immediately following recrystallization of the plaster of Paris to completely carbonize the wood flour, and then firing at a higher temperature in an oxidizing atmosphere.

Refractory brick. KIYOSI ŌTA. Japan. 131,999, Sept. 6, 1939; *Chem. Abs.*, **35**, 3053 (1941).—Iron scale or red iron ore, in such amount that the product will contain 50 to 70% Fe_2O_3, is treated with magnesia clinker and $MgCl_2$ or bittern, and the mixture is coagulated by heating, powdered, treated with aqueous $MgCl_2$ or bittern, molded, and fired.

Refractory cement. O. L. JONES (Illinois Clay Products Co.). U. S. 2,240,159, April 29, 1941 (Nov. 4, 1937).—A refractory cement for bonding silica brick comprises in combination a high-purity silica finely subdivided into varying sizes ranging from 50 mesh and smaller, a fraction of a per cent of water-retaining gum, a small amount not exceeding 10% of plastic fire clay having a medium silica content, and about 2% of bentonite to maintain the solid materials in suspension in the cement when mixed with water, the cement having a fusion point of about 3000°F.

Terra Cotta

Suitability of certain Saskatchewan clays for the manufacture of chemical stoneware. F. J. SIMPSON. *Jour. Can. Ceram. Soc.*, **9**, 43–45 (1940).—Two Saskatchewan clays were selected for study (after a consideration of known physical properties, quantity available, accessibility, and winning and transportation conditions) to ascertain their suitability for the production of good-quality chemical stoneware. All chemical stoneware used in Canada must be obtained from outside sources; this might not be advantageous during wartime. The two Saskatchewan clays tested, upon the addition of feldspar or nepheline-syenite in the correct amounts, are definitely suitable for the manufacture of chemical stoneware. Western manufacture of such a product, however, is impractical until a closer market is found. J.G.P.

Tile flooring in dwellings and public places. ANON. *Ceramica*, **2** [10] 362–70 (1940).—Tile flooring has infinite possibilities for decorative effects besides its other attri-

butes of durability and cleanliness; it is receiving interesting treatment in modern architecture. Illustrated. M.V.C.

Use of Canadian clays in engobes. E. G. PHILLIPSON. *Jour. Can. Ceram. Soc.*, 9, 50–52 (1940).—P. studied 15 engobe compositions with varying contents of kaolin, feldspar, and flint. His conclusions are as follows: (1) an increase of feldspar increases vitrification in the engobes; (2) an increase of feldspar near the high end increases crazing of the glaze; (3) low feldspar and flint in the engobes causes dullness in the glaze; and (4) high temperature increases the hardness and fit of the engobes though at the expense of the desirable white. J.G.P.

PATENT

Glazing of mosaic tile. KIYOSI YAMAMOTO. Japan. 131,584, Aug. 10, 1939; *Chem. Abs.*, 35, 3053 (1941).—Unglazed mosaic tile are arranged on a paper having lengthwise or crosswise folds, glaze is applied by spraying, the folded paper is stretched so as to separate the glazed tile from each other, and the tile are fired. This saves glaze, prevents the tile from adhering to each other, and produces uniform quality.

Whiteware

Behavior of large porcelain bushings with sudden temperature changes. HANS BARTHELT. *Elektrotech. Z.*, 62 [4] 68–72 (1941).—Thermal-shock tests were made on large porcelain bushings to obtain data on their behavior. Hot- and cold-water immersion produced stresses in the insulators, while in some cases failures due to cracks occurred. The regular test consists of immersion at 85° and 10°C., but tests were also carried out at higher temperatures (121°C.) by the use of glycerine. Failures could be detected by the sound of cracks or clinks, but the cracks were often so fine that they were invisible to the eye. When ink was painted on the surface and then washed off, a fine network of cracks could be seen, especially on the unglazed surface. Knurling or roughening of the unglazed surface with a sharp tool to provide for a cement grip was found to be a potential cause of such fine cracks, and it is assumed that porcelain in this respect acts very much like scratched glass. The mechanical destruction of insulators at this unglazed and roughened section after cementing and assembly with castings is explained as being caused by such fine invisible surface cracks in the porcelain structure. Testing by immersion in cold and hot water is harmful to heavy-wall porcelains; a test is recommended, therefore, in which an insulator is heated by an electric radiation heater to 100°C. on one side only, and the surface is then sprayed with cold water at 15°C. at an angle of 45°. This method, approximating actual field conditions, did not produce cracks in insulators, although tests were carried out to 50 cycles. This procedure is recommended in place of the present heat-shock (immersion) test. 10 illustrations. L.E.T.

Bloating and blistering of Cornish stone. A. BENNETT, A. DAWSON, J. W. WOOLISCROFT, AND H. W. WEBB. *Trans. Brit. Ceram. Soc.*, 40 [1] 14–32 (1941).—Evidence shows that bloating is due mainly to the expansion of entrapped air in the ware when it is viscous but also in part to the decomposition of gas-forming materials at the maximum temperature of vitrification. Generally speaking, a mixture of stones will cause bloating more readily than a single stone when incorporated in a pottery body. The bloating, etc., of a stone fired alone is no criterion of its behavior in a pottery body. The number of separate individual materials in a flux probably determines its bloating tendency, other things being equal. A soft flux causes bloating more readily than a hard flux. Fineness of grinding has relatively little effect on the bloating tendency of Cornish stone. R.A.H.

Causes of defects in spark-plug insulators and methods for their elimination. N. I. DIKERMAN. *Prom. Stroitel. Materialov*, 2 [9] 19–22 (1940). P.B. & E.S.

Dielectrics having high dielectric constants. K. MORITA AND S. SUZUKI. *Electrotech. Jour.* [Japan], 3 [1] 24 (1939).—Titanium insulators made of a compound of TiO_2 with additions of metallic oxides, fluxes, and mineralizers have dielectric constants more than ten times larger than those of silicate glass, steatite, mica products, and plastics, without having exceedingly high values of loss angles. The application to lenses and prisms for ultra-high-frequency waves of the decimeter order is possible. No details of the preparation of the new insulators are given. W.J.L.

Factors affecting the properties of ceramic talcose whiteware. R. F. GELLER AND A. S. CREAMER. *Jour. Research Nat. Bur. Standards*, 26 [3] 213–26 (1941); RP 1371. Price 10¢.—Talcose bodies of 12 different compositions were formed by (1) pressing, (2) hand-wedging and extrusion, and (3) hand-wedging, deairing, and extrusion. They were heated on 6 schedules: to cone 4 in 16 and 24 hr.; to cone 6 in 9, 16, and 24 hr.; and to cone 8 in 24 hr. Determinations were made of such properties as shrinkage, absorption, strength, and linear thermal expansion. Transverse-strength values showed the most pronounced differences. Deaired specimens averaged 3100 lb. per in.^{2} higher in modulus of rupture than the pressed specimens and 2300 lb. per in.^{2} higher than the hand-wedged bodies. The shrinkage was not greatly affected by the heat-treatment. The variations in heat-treatment had little effect on the thermal expansion, the averages for which ranged from 9.2 to 9.9 $\times 10^{-6}$ for the 12 bodies. R.A.H.

Heat-cycle test of suspension insulators. Y. SATOH AND K. KIMATA. *Electrotech. Jour.* [Japan], 2 [5] 120 (1938).—The method used to test suspension insulators consisted in immersing the insulators in hot water for 10 min. and then dipping them in cold water for 10 min., the difference in temperature being 60° to 80°C. After this procedure was repeated 5 times, the loss angle and equivalent leakance were determined by impressing 40 kv. at 50 cycles. The values obtained were compared with those determined before the heat cycle. Most insulators tested (commercial Japanese samples) were found to be sound in all respects. The dielectric strength of insulators which were damaged at the outer portion of the porcelain was not considerably affected. W.J.L.

Heat-shock test of large porcelain insulators. W. ESTORFF. *Elektrotech. Z.*, 62 [4] 65–67 (1941).—The heat-shock test for ceramic bodies standardized by the V.D.E. (Assn. of German Electrotechnicians) is designed to eliminate insulators which contain internal stresses developed during the firing and cooling process in ceramic kilns. Poorly fitting glazes, i.e., those having a considerably higher thermal expansion than the porcelain body, contribute greatly to such internal stresses. It has been found by experience that glazes with a somewhat lower expansion than the body are more free from stresses. In the hot-water bath (80° to 85°C.) the inside of the heavy porcelain body is under tension while the outside layer is under compression. In the cold-water bath (10° to 15°C.) the opposite situation exists. A method is described whereby a ring about 2 in. high is cut from a large cylindrical porcelain bushing. This ring is mounted in a measuring device having a micrometer gauge. The ring is then sawed through, and the distance at the slot before and after cutting through is measured. A closing-in of the ring shows that the porcelain body was under compression, while an opening toward the outside shows that the glazed porcelain was under tension. The apparatus is shown in a diagram. E. proposes this test for the present heat-shock test, which gives only empirical results. 4 illustrations. See "Testing . . . ," *Ceram. Abs.*, 20 [2] 48–49, 50–51 (1941). L.E.T.

Humidity effect on dielectric loss angles at high frequencies. S. OKAZAKI AND B. ITIJO. *Electrotech. Jour.* [Japan], 3 [9] 210 (1939).—Test pieces of Steatite B, Lowrex (borosilicate glass), and Quartzolite (polystyrene) were immersed in water for 20 hr., wiped with benzine,

and tested for the frequency characteristics of tan δ. After the pieces were heated up to 200°C. for several hours, coated with paraffin, and cooled in a desiccator, the test was repeated. After a second soaking in water for 20 hr. and wiping with benzine, the measurement was made a third time. For steatite, the effect of humidity on the leakance was especially marked in the low-frequency region. For borosilicate glass, no effect of humidity was observed. For the polystyrene, which had been dried in dry air, the effect became greater with increasing frequency. W.J.L.

Measurement of dielectric loss at ultrahigh frequencies. K. MORITA. *Electrotech. Jour.* [Japan], **3** [12] 267–71 (1939).—A modified reactance variation method is described. Using parallel Lecher wires, the method is applicable for the frequency range of the decimeter order. W.J.L.

Method of measuring high-frequency dielectric loss angles at higher temperatures. S. OKAZAKI AND B. ITIJO. *Electrotech. Jour.* [Japan], **3** [12] 287 (1939).—The resistance substitution method, using a differential condenser, was applied to the determination of high-frequency loss angles at temperatures of 100°C. and higher. W.J.L.

Porcelain-clad aluminum arresters. ANON. *Electrotech. Jour.* [Japan], **3** [4] 74 (1939).—The use of porcelain oil tanks instead of the usual steel tanks reduces the amount of insulating oil to less than 10% of that required for ordinary arresters. W.J.L.

Steatite as a high-frequency insulator (Teison No. 1). Z. KAMAYACHI AND M. SUGIURA. *Electrotech. Jour.* [Japan], **4** [10] 236 (1940).—By the addition of either pure metal oxides or pure clay, the range of allowed firing temperatures of steatite can be extended. The products (trade name, Teison No. 1) show an improvement in electrical, thermal, and mechanical properties. W.J.L.

Temperature characteristics of high-frequency dielectric loss angles. S. OKAZAKI AND B. ITIJO. *Electrotech. Jour.* [Japan], **3** [12] 287–88 (1939).—The loss angles of borosilicate glasses, steatites, and Micarex were determined up to 300°C. The lower the frequency, the steeper was the increase of tan δ with increasing temperature. At 300°C. the decrease of tan δ with increasing frequency was large for borosilicate glass, moderate for steatite, and almost negligible for Micarex. W.J.L.

Tension cracks and their prevention in hard sanitary stoneware. HEINRICH KIENBERGER. *Ber. Deut. Keram. Ges.*, **21** [11] 459–65 (1940).—A hard stoneware body developed during the last 30 years differs from ordinary stoneware as follows:

	Stoneware	Hard stoneware
Kaolin (%)	45–50	45–50
Quartz (%)	40–45	35–40
Feldspar (%)	5–10	8–15
Lime or dolomite (%)		2–5
Fired to Seger cone	1–4	7–9

Sanitary ware must be free from damage to the glaze and must be resistant to scratching by steel and attack by acids or bases. These requirements can be met, but difficulty has been caused by tension cracks which extend through both the body and the glaze. The ware may crack days or even months after firing, causing heavy loss. This cannot be avoided by design, as it is due to too high a quartz content. As quartz is the carrier of the stresses, an effort must be made not only to combine it and to keep the particles as coarse as possible but also to convert it as completely as possible wherever accessible. According to Nieuwenberg and de Nooyer, 1% calcined soda at 1300°C. for 1 hr. will convert 85% of the quartz. As soda cannot be used because of its stiffening action on the casting slip, tests were made to find a more fluid alkali frit to convert the quartz and thus to avoid the tension cracks. Almost all mixtures of alkalis, alkaline earths, and quartz give glazes with approximately the same characteristics, e.g., they all give off alkali when they come in contact with water. It would be presumptuous to claim that a certain alkali-alkaline earth-silicate, with every body used for sanitary ware, would convert the free quartz so far as to prevent tension cracks, but an example among a great number of possibilities for such a frit is as follows:

Composition	Seger formula
14.65% dolomite	0.12 CaO
44.80% quartz	0.12 MgO
23.90% calcined soda	0.69 Na_2O
16.65% feldspar	0.07 K_2O

} 0.07 $Al_2O_3 \cdot 2.70\ SiO_2$

As the fineness of the materials has considerable influence on the effectiveness of the frit, it is always best, with the help of an analysis of the slip, to keep constant conditions in experiments. The method which has made it possible for years to eliminate tension cracks completely is given. The effect of this frit begins with the addition of 0.5% and reaches a maximum at about 2%. Greater amounts should be avoided because they are apt to cause discoloration and too high a fluxing action, resulting in possible deformation of large, heavy ware. W.H.H.

To what extent can and must the ceramic industry find substitutes for pyrolusite, boric acid, and borax under the Four-Year Plan? W. FUNK. *Sprechsaal*, **72** [13] 159–61; [14] 173–76; [15] 185–86 (1939).—The uses of pyrolusite and other manganese compounds in ceramics are explained in detail. To what extent German clays or iron ores containing manganese can be used instead of pyrolusite to color pastes and engobes must be accurately determined in each case; a good color can often be obtained with substitutes, providing a small amount of pyrolusite is also added. Commercial substitutes for pyrolusite give satisfactory results in some cases. The replacement of imported raw materials by materials indigenous in Germany must be a gradual process to retain the quality of the products. Boric acid and borax may be wholly or partly omitted in the production of enamels, glazes, and decorative colored glazes in some cases. Some earthenware glazes, stoneware glazes, German porcelain glazes, and other glazes and enamels are made without boron compounds. A fritted glaze for stoneware has better properties than raw glazes where the ingredients have not been melted first. Glazes which fuse at low temperatures require some boric acid content, but by making up the glaze from several frits, the fusing point can be lowered, e.g., by combining a lead-aluminum-silicic acid frit and a boric acid frit free from lead in proportions which must be determined for every case. By this means the boric acid and lead oxide contents can be lowered. In the temperature interval between Seger cone 1*a* and 6*a* the lead oxide and boric acid contents can be greatly reduced without injuring the transparency of the glaze, and at higher temperatures they can be omitted entirely. The use of boron compounds in decorative colored glazes is discussed, and a few general suggestions are given for reducing the boric acid content. M.V.C.

Ultrahigh-frequency discharges on heat-resisting insulators. K. OKABE AND H. SEYA. *Electrotech. Jour.* [Japan], **3** [7] 167 (1939).—Waves of approximately 80 cm. were applied and discharged at the end of a thin rod electrode against heat-resisting insulators (porcelain, steatite, tydentite, hard glass, quartz, firebrick, asbestos, mica, and lava). Without preheating, surface discharge generally took place, and then, owing to dielectric loss and conduction heat, intense light was produced from the contact point followed by breakdown due to partial thermal expansion or melting of the test insulator. When the test insulator was preheated to a proper temperature, the breakdown due to partial thermal expansion was decreased, and melting generally took place. W.J.L.

Whiteware glazes free from lead and boron. W. FUNK. *Ber. Deut. Keram. Ges.*, **20** [11] 484–89 (1939).—This question became of increased importance with the outbreak of the war. In the glazes proposed by the Committee on Exchange of Raw Materials, RO is composed of CaO, MgO, ZnO, BaO, K_2O, and Na_2O; the glazes also contain suitable amounts of Al_2O_3 and SiO_2. According to Pukall (*Sprechsaal*, **51**, Nos. 37 and 38 (1918)), it was necessary

to increase the feldspar in feldspathic ware or to increase the lime in calcareous whiteware, but this procedure has disadvantages which may be overcome by adding the lime to the feldspathic ware and the feldspar to the calcareous ware. The finishing point for the former ware is lowered, perhaps from Seger cone 9 to cone 8, but for the latter it is not lowered. The glaze then consists of generally obtainable materials, such as feldspar, kaolin, sand, marble, and magnesite. Frits are completely omitted. The decrease in the firing temperature of the whiteware from cone 9 to 7 results in a considerable saving of coal. Bodies and glazes can be fired together. W.H.H.

BOOK

Testing and Evaluation of Electrotechnical Insulating Materials (Prüfung und Bewertung electrotechnischer Isolierstoffe). R. NITSCHE AND GERHARD PFESTORF. Julius Springer, Berlin, 1940. 329 pp., 190 illustrations. Reviewed in *Ber. Deut. Keram. Ges.*, **21** [6] 258 (1940).—The organic synthetic materials are treated with special thoroughness, and the ceramic insulating materials somewhat less thoroughly. Many of the methods described are equally applicable to all insulating materials. W.H.H.

PATENTS

Ceramic insulating material. K. K. POPOV AND N. V. TITOV. Russ. 54,634, March 31, 1939; *Chem. Abs.*, **35**, 3054 (1941).—From 5 to 20% (by weight) of high-grade plastic clay and 5 to 25% (by weight) of MgO, Al_2O_3, or SiO_2 are added to pyrophyllite to lower the dielectric loss.

Combination lavatory and drinking fountain. G. P. GAVIN. U. S. 2,241,194, May 6, 1941 (Oct. 26, 1940).

Crockery. W. L. GILLER. U. S. 2,239,093, April 22, 1941 (Feb. 20, 1940).—Cups, etc., of crockery having a lower portion of less external diameter than the internal diameter of the upper portion, including a plurality of circumferentially spaced shoulders on the upper portion, whereby when the article is fitted into another similar article the shoulders rest on the brim of the other article and define intermediate axially directed spaces opening through the lower edge of the article for the admission and escape of washing or sterilizing fluid into and from the space between the articles.

Dripless spout. CHRISTOPH ACHTZIGER (Porzellanfabrik Weiden Gebr. Bauscher Zweigniederlassung der Lorenz Hutschenreuther A.-G. Selb.). U. S. 2,241,840, May 13, 1941 (Aug. 23, 1938).

Insulator. W. H. BURLESON (Ohio Brass Co.). U. S. 2,239,809, April 29, 1941 (March 9, 1939).

Manufacture of metal-to-porcelain seals. M. J. CUTTLER. U. S. 2,241,505, May 13, 1941 (Aug. 21, 1936).

One-fire method of manufacturing ceramic articles. H. R. GOODRICH (Gladding, McBean & Co.). U. S. 2,241,705, May 13, 1941 (Nov. 5, 1938).—The method of making in a single firing a ceramic unit comprises forming a mixture consisting of a material containing at least 35% of the minerals selected from the group consisting of calcium and calcium magnesium-containing silicates substantially free of volatile content tending to disrupt during firing the subsequently formed structure, the minerals being characterized by a fibrous compact bladelike structure, and a clay binder therefor, the mixture being substantially free of extraneously added fluxing materials, pressing and compacting the mixture into hard plates and granules, disintegrating the latter, pressing and shaping the disintegrated mixture, coating the shaped article with an intermediate protective coating, applying a glaze upon the coating, and maturing the article including the glaze at one firing at the glost temperature.

Spark plug. BENDIX AVIATION CORP. Brit. 535,426, April 23, 1941 (Oct. 7, 1938).

Equipment and Apparatus

Agitation: mass transfer coefficients in liquid-solid agitation systems. ARTHUR W. HIXSON AND SIDNEY J. BAUM. *Ind. Eng. Chem.*, **33** [4] 479–85 (1941).—An approximate integrated form of the cube-root law is derived. This equation is used to determine mass transfer data for several liquid-solid combinations in a series of dimensionally similar agitators. For any particular liquid-solid system under isothermal conditions, a logarithmic plot of the dissolution constant, K, against the product of the speed and the size factors, nd, yields a straight line. The results for several systems can be correlated on a dimensional basis. Two distinct regimes of flow are noted. Above a Reynolds number of 6.7×10^4, the data are represented by

$$\frac{Kd}{D} = 0.16\left(\frac{nd^2\rho}{\mu}\right)^{0.62}\left(\frac{\mu}{\rho D}\right)^{0.5}$$

and below this value by

$$\frac{Kd}{D} = 2.7 \times 10^{-5}\left(\frac{nd^2\rho}{\mu}\right)^{1.4}\left(\frac{\mu}{\rho D}\right)^{0.5}.$$

For design purposes, the method of correlation is more general than that previously proposed by Hixson and Wilkens. Illustrated. F.G.H.

Apparatus for the absorption or gravimetric determination of constituents of a gas mixture. MARTIN SHEPHERD AND HARRY W. BAILEY. *Jour. Research Nat. Bur. Standards*, **26** [4] 347–49 (1941); RP 1381. Price 5¢.—The apparatus described eliminates many of the undesirable features of certain types of "absorption trains" which are intended to remove constituents from gas mixtures or to determine them gravimetrically. It is adaptable to many uses, since auxiliary units, such as combustion apparatus, may be connected by means of interchangeable joints. R.A.H.

Application of the schlieren method to instruments heated by an electrical source. S. SHIMIZU, T. INAI, AND I. NISHIFUJI. *Electrotech. Jour.* [Japan], **4** [1] 22 (1940).—A simple arrangement for the application of the schlieren method to the study of the flow of heat on insulators and other electrically heated materials is described. W.J.L.

Deairing clay by a vacuum process. OTTO MANFRED. *Argile*, No. 195, pp. 17, 21 (1939).—The improved helix press is in reality three machines in one: stoker, vacuum mixer, and molding press. The necessary degree of mixing, wetting, and air elimination from the mass is accomplished, and the mass is then molded in the press without defects and fissures. See *Ceram. Abs.*, **20** [5] 123 (1941). M.V.C.

Device for measuring the temperature of surfaces. ANON. *Glashütte*, **71** [2] 20 (1941).—For accurate measurement of the surface of masonry or the outer wall of a cooling canal, a metal case of copper, brass, iron, or other good heat-conducting metal, with a base or foot which is set in the surface to be measured, was devised. A thermometer is placed in the case, and the space around it is filled with mercury to give an all-metal contact. Illustrated. M.V.C.

Direct-reading measuring microscope. J. E. SEARS AND A. TURNER. *Jour. Sci. Instruments*, **18** [2] 17–19 (1941).—A traveling microscope for measuring Debye-Scherrer X-ray powder photographs is described. Measurements are made by direct reference to a glass scale which can be traversed under the microscope with both scale and film together in the field. A fixed vernier of glass is viewed at the same time to give readings to 0.01 mm. 6 figures. J.L.G.

Electric-furnace manufacture. ANON. *Mech. World & Eng. Record*, **109** [2826] 147 (1941).—The factory of Wild-Barfield Electric Furnaces, Ltd., and its testing plant and laboratory are briefly described. F.E.S.

Electric heating mortar for use in carbon and hydrogen microcombustions. G. FREDERICK SMITH AND W. H.

TAYLOR. *Ind. Eng. Chem., Anal. Ed.*, **13** [3] 203–204 (1941).—Details of construction and operation are given. Illustrated. F.G.H.

Electron microscope. A. T. WESTERMAN. *Amer. Jour. Sci.*, **239** [5] 386 (1941).—The fundamental limitation of the optical microscope lies in the character of light, i.e., its wave length, as a train of light waves will form a clear image only when the dimensions of the object are greater than the wave length, which lies between 0.00004 and 0.00007 cm. for visible light. This limit of the resolving power of a microscope may be improved through the use of ultraviolet light; glass is opaque to these wave lengths, however, and as the wave length becomes shorter it becomes more difficult to obtain materials that are transparent. For these reasons, the useful magnification of microscopes using visible light is limited to about 1500 diameters, while with ultraviolet light a magnification of about 2500 diameters may be obtained. In an electron microscope the electrons are set in motion by an electric field, and the wave length may be varied according to the voltage applied. Electrons subjected to a voltage of 100,000 volts have a wave length of 4.3×10^{-10} cm. which is about 100,000 times smaller than the shortest usable waves of ultraviolet light. The beam of electrons can be made to diverge or converge as desired by passing them through holes in positively charged plates of metal and through current-carrying coils, respectively. The electrons can be made to produce a visible image by allowing them to strike a fluorescent screen. Glass slides cannot be used, as the electrons will not penetrate material of any considerable thickness. A thin film of collodion may be substituted. A direct magnification of 25,000 diameters may be obtained. Photographs of the image may be further magnified to yield a total magnification of 100,000 diameters. B.C.R.

Judgment on the superiority of mica by means of the schlieren method. S. SHIMIZU AND I. NISHIFUJI. *Electrotech. Jour.* [Japan], **4** [10] 231–32 (1940).—The schlieren method reveals differences in the quality of mica samples by making visible minute defects (rust, air bubbles, etc.) which are responsible for lowering the breakdown limit and heat resistance. W.J.L.

Limitations of diffusion equations in drying. O. A. HOUGEN, H. J. MCCAULEY, AND W. R. MARSHALL, JR. *Trans. Amer. Inst. Chem. Engrs.*, **36**, 183–209 (1940).—The limitations in the application of diffusion equations to drying are pointed out. In the drying of solids, the movement of liquid water by diffusion is restricted to the equilibrium moisture content below the point of atmospheric saturation and to single phase solid systems in which the water and solid are mutually soluble. The first category applies to the last stages in the drying of clays, starches, flour, textiles, paper, and wood, and the second category applies to the drying of soaps, glues, gelatins, and pastes. The differential diffusion equations can be applied to calculate the distribution and rates of drying under the conditions cited above, but integrations should allow for the variable nature of diffusivity. Water held in the interstices of solids, as liquid covering the surface, and as free water in cell cavities is subject to movement by gravity and capillarity, provided passageways for continuity of flow are present. Water vapor may be removed by vapor diffusion through the solid, provided a temperature gradient is established by heating which creates a vapor-pressure gradient toward the drying surface. Vaporization and vapor diffusion may be applied to any solid where heating takes place at one surface and drying from the other and also where water is isolated between granules of solids. Diffusion equations can be properly used only with due regard to the actual mechanism of liquid movement and its complex nature. H.E.S.

Manometric gas-analysis apparatus. MARTIN SHEPHERD AND E. O. SPERLING. *Jour. Research Nat. Bur. Standards*, **26** [4] 341–46 (1941); RP 1380. Price 5¢.—The apparatus described is designed for the analysis of gas mixtures when (1) the volume of the sample is small (about 5 to 0.5 ml.), (2) the volume of the sample is of the order of magnitude of 0.2 to 0.1 ml. and an approximate microanalysis is desired, and (3) the gases to be analyzed are dissolved in some liquid from which they must be liberated prior to analysis. R.A.H.

Measurement of flow properties with the Gardner mobilometer. PAUL W. KINNEY. *Ind. Eng. Chem., Anal. Ed.*, **13** [3] 178–85 (1941).—To obtain the true value of the force overcoming the viscous or plastic resistance of the material, a buoyant-force correction must be subtracted from the weight producing the shear. This correction is especially important where the shearing weight is relatively small. The instrument can be calibrated with a standard liquid, so that the results can be given in absolute units by the following equations: $\eta = K(W - 6\rho)t$ for a true liquid; $1/t = \mu(KF - Kf)$ for a plastic soft solid. Illustrated. F.G.H.

Multipurpose photoelectric reflectometer. R. S. HUNTER. *Jour. Research Nat. Bur. Standards*, **25** [5] 581–618 (1940); *Jour. Optical Soc. Amer.*, **30**, 536–59 (1940).—The reflectometer was developed to measure apparent reflectance, specular gloss, and trichromatic coefficients. These measurements are useful in the ceramic, paint, textile, paper, and chemical industries to indicate the lightness, gloss, and color of finished articles. Two light beams from a single source are directed along separate paths to two barrier-layer photocells. A galvanometer indicates equality of the currents generated by the two cells. For each test the photometric adjustments which restore equality of the currents are measured on direct-reading scales, one for apparent reflectance and the other for specular gloss. The instrument is suitable for measuring small differences in the apparent reflectance, gloss, or color of nearly identical samples. A.P.

New instrument for rheological studies of plastic substances. C. R. BAILEY. *Ind. Eng. Chem., Anal. Ed.*, **13** [3] 173–77 (1941).—B. discusses the aims, details of construction, accessories, and procedure of a new instrument for rheological studies of plastic substances. F.G.H.

New type of wide aperture ratio telescope objective. A. E. GLANCY. *Jour. Optical Soc. Amer.*, **30**, 572 (1940).—The typical achromatic telescope objective is preceded by a zero-power meniscus lens. The resulting objective is achromatic, is free from spherical aberration and coma, and has a wide useful aperture. The principle may be applied to photographic lenses, projection systems, and microscopes. A.P.

Optical properties of colloidal suspensions in relation to the measurement of particle-size frequency. E. G. RICHARDSON. *Jour. Applied Physics*, **11**, 653–57 (1940).—An apparatus has been developed for deriving the size-frequency curve in the 0.1- to 1-micron range from light transmission through a polydisperse suspension during sedimentation. Results were obtained for titanium oxide, sulfur, clay, and zinc oxide. A.P.

Optical pyrometers. S. N. DUGUID. *Steam Engr.*, **9**, 365–67 (1940).—Wanner and disappearing-filament types of optical pyrometers are described, and their use is explained. L.R.B.

Orifice nomograph. D. S. DAVIS. *Ind. Eng. Chem.*, **33** [3] 420 (1941). F.G.H.

Particle-size determination by sedimentation. KARL KAMMERMEYER AND J. L. BINDER. *Ind. Eng. Chem., Anal. Ed.*, **13** [5] 335–37 (1941).—Details of construction and operation are given for an improved type of apparatus. Illustrated. F.G.H.

Precision of microchemical balances. MARY CORNER AND HAROLD HUNTER. *Analyst*, **66** [781] 149 (1941). B.C.R.

Preparation of high-melting alloys with the aid of electron bombardment. R. HULTGREN AND M. H. PAKKALA. *Jour. Applied Physics*, **11**, 643–46 (1940).—An electron-bombardment furnace was constructed for high-temperature melting at a cost of $500. The temperature attainable is limited only by the refractoriness of the crucible. See "High-temperature . . . ," *Ceram. Abs.*, **18** [6] 156 (1939). A.P.

Screen color-mixing methods. V. H. REMINGTON. *Glass Ind.*, **22** [4] 154–56 (1941).—Color mixing involves

coating each individual particle of impalpable powder with oil. Equipment has evolved from hand mullers to iron paint, colloid, and three roll paint mills. The latter is recommended. Preliminary mixing of dry color and squeegee oil in a pony mixer is favored. A.P.

Silica and Pyrex brand glass stills with automatic constant-pressure feeds. C. S. PIPER AND A. C. OERTEL. *Ind. Eng. Chem., Anal. Ed.*, **13** [3] 191–92 (1941).—Details of construction and operation are given. Illustrated. F.G.H.

S.I.L. viscometer. E. L. RUH, R. W. WALKER, AND E. W. DEAN. *Ind. Eng. Chem., Anal. Ed.*, **13** [5] 346–49 (1941).—A kinetic viscometer has been developed and proved practical in actual routine testing operations. It is almost, if not fully, as satisfactory as a more expensive patented instrument of foreign origin which has been extensively used. Illustrated. F.G.H.

Simple method for refractive-index determinations. S. M. COX. *Jour. Sci. Instruments*, **18** [2] 20–21 (1941).—C. describes an instrument and a method for measuring the refractive index of glass rods to an accuracy of about 0.5%. No grinding or polishing other than a fire polish is necessary. 1 figure. J.L.G.

Standard screens. R. H. FIELD. *Jour. Can. Ceram. Soc.*, **9**, 9–13 (1940).—The necessity for standards in relation to screens is dictated by the same considerations governing other standards, i.e., the establishment of a reference medium permitting measurements made at different times and by different observers to be accurately correlated. F. discusses various standards in existence, presents specifications for testing sieves adopted by the Canadian Government Purchasing Standards Committee, and gives a table showing equivalent sieves in different series. J.G.P.

Viscosity measurement. M. R. CANNON AND M. R. FENSKE. *Ind. Eng. Chem., Anal. Ed.*, **13** [5] 299–300 (1941).—The authors describe a modification of the Ostwald viscometer which is particularly suited for measuring the viscosities of opaque liquids. Illustrated. See *Ceram. Abs.*, **18** [1] 29 (1939). F.G.H.

SEPARATE PUBLICATION

Ni-Hard *versus* Abrasion. Mond Nickel Co., Ltd., London, 1940. 24 pp. Free.—This pamphlet contains reprints of papers on this subject, including "Ni-Hard in clayworking machinery" (*Ceram. Abs.*, **19** [1] 21 (1940)). See also "Specially . . . ," *ibid.*, **20** [4] 99 (1941). L.R.B.

PATENT

Wet concentration of kaolin. A. F. NISHCHENKOV. Russ. 54,395, Jan. 31, 1939; *Chem. Abs.*, **35**, 3053 (1941).—The pulp is passed through a trough, and the fraction that precipitates is separated by means of a scraper conveyer submerged in the pulp and moving in the same direction as the pulp to eliminate frothing.

Kilns, Furnaces, Fuels, and Combustion

Central gas generator. O. EHINGER. *Glashütte*, **71** [6] 68–69 (1941).—A centrally situated gas generator with overhead gas conduits to the furnace or place of consumption has been found more economical than any other system. Only one reserve generator is required. With good insulation of conduits, some of the heat of the gas coming from the generator is retained. A design of a plant with a central gas generator is described and illustrated. M.V.C.

Coal hydrogenation. Natural bituminous coal; artificial cellulose coal and its bitumen and residual portion. E. BERL, H. BIEBESHEIMER, AND W. KOERBER. *Ind. Eng. Chem.*, **33** [5] 672–74 (1941). F.G.H.

Economical pot and tank furnaces. E. S. *Glashütte*, **71** [1] 7 (1941).—For economical firing of pot and tank furnaces, the condition of the fuel which is to be converted into gas is important; it should not be weathered or wet if the maximum yield of gas is to be obtained. Especially porous fuel should be covered during transportation and storage to prevent the absorption of water, which must be evaporated before gasification can take place. To evaporate 1 kgm. of water frozen in fuel, 716 heat units are required; 1 kgm. of liquid water requires 637 heat units. Hot dry weather is injurious because it tends to weather the coal, as does long storing. Fuel that is too wet or weathered has a reduced gas yield, and more fuel is therefore required. The "pit moist" condition is considered the best condition in which to gasify coal. M.V.C.

Flame temperature. BERNARD LEWIS AND GUENTHER VON ELBE. *Jour. Applied Physics*, **11**, 698–706 (1940).—The theoretical flame temperature corresponding to complete statistical equilibrium in the burned gas is distinguished from the experimental flame temperature measured by the gas law equation and corresponding to equilibrium in translational degrees of freedom. The latter may be higher due to excitation lag in the internal, presumably vibrational, degrees of freedom. The technique of measuring flame temperatures by spectral line-reversal, wires, and thermocouples is described. Soot-forming luminous flames may be regarded as in complete statistical equilibrium, the temperature difference between soot particles and the surrounding gas being negligible. Methods of determining the temperature and emissivity of such flames are described. In industrial furnaces, the differences between experimental and theoretical temperatures should be negligible. A.P.

Free energies of formation of gaseous hydrocarbons and related substances. C. M. THACKER, H. O. FOLKINS, AND E. L. MILLER. *Ind. Eng. Chem.*, **33** [5] 584–90 (1941).—Standard free energies of formation have been calculated for 77 hydrocarbons and 8 inorganic compounds in the gaseous state at 100° intervals from 298.1° to 1200°K. The tables include values for the normal paraffins (methane to *n*-decane), the 1-olefins (ethylene to 1-decane), various branched-chained saturates and unsaturates of 4 to 8 carbon atoms, and the inorganic compounds carbon monoxide, carbon dioxide, water, ammonia, carbon disulfide, hydrogen sulfide, sulfur dioxide, and sulfur trioxide. Distinction is made between the more accurate values for straight-chain hydrocarbons and those for their branched-chain isomers. Illustrated. F.G.H.

Graphs for gas flues and chimneys of continuous kilns. K. SPINGLER. *Tonind.-Ztg.*, **63** [69] 785–87 (1939).—S. attempts for the first time the graphical determination of data for the firing of continuous brick kilns by using diagrams such as those long known for calculating data for boiler furnaces. One graph shows the air ratio in relation to the calorific value of the coal and for various multiples of excess air, another gives the velocity of flue gases as a function of the gas volume and the flue cross-section area, and a third diagram deals with the draft as a function of the height of the chimney at various temperatures. F.E.S.

Liquid-vapor equilibrium relations in binary systems: *n*-butane–*n*-heptane system. W. B. KAY. *Ind. Eng. Chem.*, **33** [5] 590–94 (1941).—The *P-V-T-x* relations at the liquid- and vapor-phase boundaries in the *n*-butane–*n*-heptane system were worked out from measurements on a series of mixtures varying in composition from pure *n*-butane to pure *n*-heptane. *T-x* diagrams of the coexisting liquid and vapor at constant pressure were constructed, from which data were obtained for calculating the phase-equilibrium constants. Information on the effect of the physical properties of the mixture on the phase-equilibrium constants is given by a comparison of the constants for *n*-butane dissolved in ethane and in *n*-heptane and of those for *n*-heptane dissolved in ethane and in *n*-butane. Illustrated. F.G.H.

Modern gas-fired furnaces. E. MENZEL. *Werkstatt & Betrieb*, **73** [9] 200–202 (1940).—Provided insulation is

adequate and preheated air is used, modern gas furnaces are equivalent to electric furnaces. W.J.L.

Natural gas hydrates. WILLARD I. WILCOX, D. B. CARSON, AND D. L. KATZ. *Ind. Eng. Chem.*, **33** [5] 662–65 (1941).—Natural gases under pressure have been known to form crystalline hydrates with water. Data are given on the temperatures and pressures, up to 4000 lb. per sq. in., at which three natural gases form hydrates. The phase data on hydrates of pure hydrocarbons were enlarged by the equilibrium temperatures and pressures for the hydrate formation from compressed liquid propane. The concept was introduced of handling vapor-solid equilibria in the manner in which the vapor-liquid equilibria data have been used to predict the phase relations of fluid hydrocarbon systems. Illustrated. F.G.H.

Nomographs for thermal conductivities of gases and vapors. D. S. DAVIS. *Ind. Eng. Chem.*, **33** [5] 675–78 (1941). F.G.H.

Recent automatic draft regulation in ceramic kilns and automatic control of gas pressure. C. KABUS. *Ber. Deut. Keram. Ges.*, **21** [6] 237–55 (1940).—The draft depends on the pressure, temperature, and density of the exhaust gas, the density varying with the composition and temperature of the gas. Instruments are necessary to measure the draft and to keep it as constant as possible. A draft may be produced artificially by ventilators, but a chimney is more economical in the long run. With the most accurate draft regulators, there is a measuring system which acts not directly on the damper but indirectly through a relay. Illustrations are given of such a regulator and of the autographic record of hardly perceptible variations in draft when it is installed. In the deflecting plate gas-pressure regulator, the relay acts in only one direction, the return motion being due to a weight. Other regulators which make use of a float, diaphragm, or the Venturi principle are described and illustrated. W.H.H.

Temperature coefficient of density and refractive index for hydrocarbons in the liquid state. M. R. LIPKIN AND S. S. KURTZ, JR. *Ind. Eng. Chem., Anal. Ed.*, **13** [5] 291–95 (1941).—Using the equation $dt/4 = d20/4 + \alpha(t - 20) + \beta(t - 20)^2$, a single curve of α vs. molecular weight and a single curve of β vs. molecular weight were found applicable to all types of hydrocarbons with very few exceptions. From these curves, the temperature coefficient of density of a liquid hydrocarbon at atmospheric pressure can be predicted from the molecular weight within 0.00002 gm. per cc. per °C. The only definite exceptions to this relation are benzene, pentaethylbenzene, and hexapropylbenzene, which show coefficients 0.00010 gm. per cc. per °C. higher than the predicted values. The curves are also applicable to hydrocarbon mixtures. This relation is more general than any previously published for predicting the temperature coefficient of density. The corresponding relationship between compressibility and molecular weight is being investigated. Since Ward and Kurtz have shown that, for hydrocarbons, $\Delta n = 0.60\ \Delta d$, an accurate method of calculating density change also provides a satisfactory method for calculating change in refractive index. Illustrated. F.G.H.

Geology

Age measurements by radioactivity. CLARK GOODMAN AND R. D. EVANS. *Bull. Geol. Soc. Amer.*, **52** [4] 491–544 (1941).—The radioactive methods of geologic-age determination involve four basic requirements: (1) a known systematic rate of disintegration of the radioactive elements, (2) accurate measurement and sampling, (3) absence of the disintegration product as a primary constituent, and (4) no addition or subtraction of the disintegration product or its source during the history of the material. A critical review is presented of the fulfillment of these requirements by the various radioactive methods. Emphasis is placed on the helium method because of its possible wide applicability, because no adequate review has heretofore appeared, and because considerable confusion exists concerning the present status of this method. Conclusions are as follows: (1) The retentivity of rocks for helium is the major source of uncertainty in the present application of the helium method. (2) Analytical errors, earlier misconceptions, and the lack of attention to the basic requirements have necessitated the discarding of much of the age research of the past three decades. (3) Isotopic abundance measurements are essential in the determination of ages by the lead method. (4) More specific geological dating is needed in the formulation of a useful lead time scale. (5) More direct comparisons between the lead and helium age ratios are urgently needed. (6) Although other methods have been discovered, the lead and helium methods still remain the outstanding promising radioactive methods of quantitative age determination. (7) Recent developments in the application of the helium method to selected common rock minerals promise a method of dating igneous rocks that should be of wide application as an aid to geologic correlations. See "Radioactivity . . . ," this issue, p. 180. A.C.B.

Beryllium and beryl in the ceramic industry. L. GUIMIN. *Rev. Matériaux Construction Trav. Publics*, No. 359, pp. 85–87B (1939); see "Glucinium . . . ," *Ceram. Abs.*, **17** [3] 115 (1938). M.V.C.

Chemical investigation of silicates: X, Topaz, $Al_2SiO_4(F,OH,O)_2$, and its synthesis and a new aluminum oxide containing fluorine. ROLF SCHOBER AND ERICH THILO. *Ber. Deut. Chem. Ges.*, **73** [11] 1219–40 (1940).—Analytical and X-ray investigations of three natural topazes showed no simple relation between fluorine content and lattice value. Thermal hydrolysis of AlF_3 yields aluminum oxyfluoride of the approximate composition Al_2O_3F or α-Al_2O_3, depending on the temperature. Up to 600°C. only oxyfluoride is formed, from 600° to 800°C. oxyfluoride with α-Al_2O_3, and above 800°C. pure α-Al_2O_3. X-ray examination of the oxyfluoride shows that this substance has its own specific lattice. The intensities of the lines differ from the Debye diagrams according to whether the fluoride is prepared at 550° or at 600°C. Very sharp temperature limits were shown for the reaction $2AlF_3 + 2SiO_2 =$ topaz $+ SiF_4$. The formation temperature of topaz is between 750° and 950°C. Below and above these temperature limits a material is formed, with a heavy loss of SiF_4, whose powder photographs show a lattice similar to that of mullite. This material contains 5 to 6% F. Experiments with an excess of SiO_2 or AlF_3 confirm the reaction mechanism for the formation of topaz by the following reactions: $4AlF_3 + 6H_2O = 2Al_2O_3 + 12HF$; $12HF + 3SiO_2 = 3SiF_4 + 6H_2O$; $2Al_2O_3 + SiF_4 + SiO_2 = 2Al_2SiO_4F_2$. Experiments in which topaz was formed by the reaction of SiF_4 on Al_2O_3 or on a mixture of $2Al_2O_3 + 1SiO_2$ showed that for all these reactions the same temperature limits exist. It is possible to obtain topazes with widened lattice; for this the Al_2O_3 must be present in the especially "active" form. In the temperature region up to 1050°C., no mullite-like material is formed by the reaction of SiF_4 with $Al_2O_3 + SiO_2$. This indicates that the mullite formation from AlF_3 and SiO_2 does not occur in the manner of the topaz formation, but in some other way, probably by a reaction in the solid state. This also explains the great difference in the speed of formation of the topaz and this mullite-like material. **XI, Further experiments with pyrophyllite and a comparison of its reactions with those of talc ($Mg_3[Si_4O_{12}](OH)_2$).** ERICH THILO AND ULRICH SCHWARZ. *Ibid.*, **74** [2] 196–204 (1941).—Pyrophyllite ($Al_2[Si_4O_{10}](OH)_2$), in the absence of foreign material, changes into anhydrous pyrophyllite with the loss of 1 mol. of water: $Al_2[Si_4O_{10}](OH)_2 = Al_2[Si_4O_{10}]O + H_2O$. Beginning at 1150°C., this dehydrated pyrophyllite decomposes to form mullite and amorphous silica which at 1200°C. changes into cristobalite: $3Al_2[Si_4O_{10}]O \longrightarrow 3Al_2O_3 \cdot 2SiO_2 + 10SiO_2$. The same decomposition occurs in the presence of foreign

material, but the foreign materials cause a considerable drop in the mullite and cristobalite formation temperature. At about 800°C. pyrophyllite and anhydrous $MgCl_2$ form cordierite, SiO_2, and HCl according to the following reaction: $2Al_2[Si_4O_{10}](OH)_2 + 2MgCl_2 = Mg_2[Al_3(AlSi_3)O_{18}] + 3SiO_2 + 4HCl$. With ZnO (at 1000°C.), zinc spinel, zinc orthosilicate, and water are formed: $Al_2[Si_4O_{10}](OH)_2 + 9ZnO = ZnAl_2O_4 + 4Zn_2SiO_4 + H_2O$. Pyrophyllite and MgO (mol. ratios 1:5, 1:10, and 1:1) and cordierite and MgO were investigated at temperatures between 800° and 1200°C., and the resulting compounds, such as olivine, spinel, cordierite, and mullite, are described. Other experiments with pyrophyllite and CuO gave copper spinel plus SiO_2 at 950°C., while cobalt ($CoCl_2$) with pyrophyllite gave Co spinel and SiO_2 at 800°C. It appears that the dissociation temperature of the pyrophyllite and the formation temperature of the crystallized SiO_2 modifications may be considerably lowered by the introduction of suitable foreign materials. It also appears that the transformation temperature of a silicate is less dependent upon the kind of Si–O–Si chains than upon the nature of the added material. Oxides (and chlorides), MgO, $MgCl_2$, $CoCl_2$, and CuO, are especially suitable. The results obtained by Parmelee and Barrett (*Jour. Amer. Ceram. Soc.*, **21** [11] 388–93 (1938)), who found that WO_3 and V_2O_5, but not B_2O_3 and P_2O_5, have the property of lowering the dissociation temperature of pyrophyllite and the transformation temperature of cristobalite, tend to confirm this theory. The well-known use of K_2CO_3 and Na_2CO_3 for the digestion of silicates rests upon the same principle. For Part **IX** see *Ceram. Abs.*, **19** [3] 77 (1940). L.E.T.

Control of clays containing varying quantities of adsorbed salts. C. M. LAMBE, JR. *Bull. Amer. Ceram. Soc.*, **20** [5] 155–58 (1941).—4 figures.

Cooling of the earth. L. B. SLICHTER. *Bull. Geol. Soc. Amer.*, **52** [4] 561–600 (1941).—Ancestral molten earth is assumed. The earth now consists of a heavy core with a radius of about 3400 km., a thick lighter solid mantle, and a thin crust. The mantle probably solidified from the bottom up with heat transported by convection currents. The earth has a large heat capacity. Thermal equilibrium is probably far from achieved. Surface heat flows fix definite upper limits for the amount of radioactivity which may exist within 100 to 200 km. of the surface. No information is obtainable by thermal observations or theory about the thermal state below 200 to 300 km. in depth, and it is therefore unknown whether the earth is heating or cooling. A.C.B.

Dressing of Palaboroa vermiculite. J. E. LASCHINGER. *Jour. Chem. Met. Mining Soc. S. Africa*, **40** [6] 213–26 (1939).—Vermiculite occurs in South Africa at Palaboroa in the northeastern Transvaal associated with calcrete and apatite in massive pyroxenite. It is more easily separated by crushing and winnowing through an air blast than by the dry-tabling methods used in the U. S. The air blast may be easily arranged in such a way that the thickest reasonably clean vermiculite is just slightly thicker than that giving equidimensional grains after expansion. Grains thicker than this offer no technical or commercial advantages. See "Vermiculite Deposits . . . ," *Ceram. Abs.*, **19** [1] 28 (1940). L.W.V.

Electrooptical field mapping. H. MUELLER. *Jour. Optical Soc. Amer.*, **31**, 286–91 (1941).—As a result of the discovery of an unusually large electrooptical (Kerr) effect in certain colloidal solutions of bentonite, it has become possible to study and measure inhomogeneous electric fields by an optical method which is analogous to the photoelasticity method. A.P.

Factors influencing flocculation and precipitation—theories and variables. E. M. BEAVERS, J. E. MAGOFFIN, AND F. K. CAMERON. *Textile Research*, **11**, 23–29 (1940); *Chem. Abs.*, **35** [2] 365 (1941).—Flocculation and precipitation of a colloidal dispersion are inseparably related inasmuch as the phenomena associated with the latter process are closely dependent upon those of the first. Information relevant to both processes and to the variables encountered in such an investigation is discussed briefly. 40 references.

Flotability of some minerals. M. MARTENSON. *Tids. Kjemi Bergvesen*, **20**, 99–100 (1940); *Chem. Abs.*, **35** [2] 346 (1941).—The flotation properties of some pegmatitic minerals were determined and classified according to a simple index system. It appears that it should be possible to float a number of valuable pegmatitic minerals from quartz-feldspar-mica "ore."

Fundamental study of clay: II, Mechanism of deflocculation in the clay–water system. A. L. JOHNSON AND F. H. NORTON. *Jour. Amer. Ceram. Soc.*, **24** [6] 189–203 (1941).—23 figures, 59 references. For Part I see *ibid.*, [2] 64–69.

Geologic temperature recorders. N. L. BOWEN. *Sci. Monthly*, **51** [1] 5–14 (1940).—The temperatures of magmas (molten rock) have seldom if ever exceeded 1200°C. when they have arrived in that part of the crust of the earth which becomes accessible to observation as a result of erosion. Most magmas have probably not exceeded 900°C., and some, especially those rich in alkali-aluminous silicates, have had temperatures little in excess of 600°C. The temperatures of consolidation (crystallization) of the igneous rocks formed from these magmas are only slightly lower than the temperatures of the magmas themselves, i.e., the magmas carry little superheat. The temperatures of crystallization are highest for rocks rich in lime-magnesian constituents and lowest for those rich in alkali-aluminous silicates and free silica. The crystallization temperatures of pegmatites, which are formed from residual magmas especially rich in water and other volatile substances and are among the storehouses of the radioactive elements, lie in the neighborhood of 573°C. Mineral veins formed from the residual aqueous solutions after crystallization of the main rock constituents were deposited at temperatures in part approaching those of pegmatites and extending from this range down to the temperature of boiling water and even lower, with characteristic minerals at each stage. There are, in addition, rocks and mineral deposits formed under conditions attended by rising temperatures. Sediments and other materials formed at the surface of the earth at prevalent temperatures may be buried under later accumulations; as a result of such burial they experience a rise in temperature. New minerals sometimes form, the original minerals invert to high-temperature forms, and some assemblages probably suffer a certain amount of selective fusion. The extreme of reheating is experienced when such materials are immersed in molten magmas, in which case the more susceptible are themselves melted. Indications are, however, that even under these most extreme conditions rock materials have probably never been heated above 1150°C. H.E.S.

Helium retention in common rock minerals. P. M. HURLEY AND CLARK GOODMAN. *Bull. Geol. Soc. Amer.*, **52** [4] 545–60 (1941).—By the helium method, the age of material is determined by the ratio of the helium content to the content of radioactive elements. Significant disparities were found in minerals from unaltered Triassic diabase. Pyroxene from six samples gave a mean age ratio of 103 million years, and feldspar from the same samples gave age ratios of 36 million years. Six samples of late Triassic magnetite gave age ratios closely grouped around a mean value of 134 million years, despite widely varying contents of radium and thorium; thus magnetite has a high helium retentivity. This conclusion is supported by magnetite specimens of various geological ages. A.C.B.

High-frequency characteristics of rocks. M. KADOWAKI. *Electrotech. Jour.* [Japan], **3** [6] 143 (1939).—K.'s purpose was the collecting of data for prospecting by means of high-frequency electric measurement. The resistivity and the dielectric constants of (1) red chert, (2) fine-grained biotite, (3) fine-grained sandstone, and (4) serpentine ore were determined. The effective resistances, determined by means of the resistance insertion method, increased in the order $4 < 3 < 2 < 1$. The dielectric constants, determined by the compensation method, decreased in the order $4 > 3 > 2 > 1$. The values of the resistance at 6 m.

were found to amount to only $^1/_{10}$ of those at 200 m. The values of the dielectric constants began to decrease considerably in the region of 80 to 40 m. *Ibid.*, [9] 208–209.—The resistivities and dielectric constants within the range of wave lengths between 200 m. and 5 m. were determined for dry and moisture-containing minerals. Limestone, crystalline schist, pyroxene andesite, vein quartz bearing argentite, chert, hornblende granite, calcite, fine-grained biotite granite, biotite-hornblende gneiss, epidote schist, marble, graphite schist, diorite, chlorite schist, tuffaceous sandstone, chromite, shale, fine-grained sandstone, and serpentine were tested. The high-frequency resistivity of dry samples was considerably different from the d.-c. or low-frequency a.-c. resistivity, and at low values it decreased with decreasing wave length. The dielectric constants of the dry samples showed a tendency to decrease with decreasing wave length, and this decrease was very rapid in the region of ultrashort waves. When a small quantity (less than 1% in weight) of evaporated water had been added to the dry minerals, the resistance was considerably lowered but the dielectric constant increased. W.J.L.

Increasing plasticity and dry strength of flint and semi-flint clays by treatment with electrolytes. E. B. Snyder and R. M. King. *Bull. Amer. Ceram. Soc.*, **20** [5] 151–53 (1941).—2 references, 1 figure.

Investigations of clay. Microflora and the physico-mechanical properties of clays under various conditions of maturation. R. B. Angenitskaya, M. D. Bogopolskiĭ, O. F. Drako, and N. M. Pidoplichka. *Geol. Zhur. Ukraïn. Akad. Nauk*, **6** [1–2] 305–22 (in English, 317–18) (1939); *Chem. Abs.*, **35** [2] 590–91 (1941).—Microorganisms and hydrolytic processes stimulate chemical transformations in clays, leading to the formation of molecularly dispersed substances and an improvement in plastic properties. During maturing, Fe and Ca compounds are reduced, while H_2O, salts, N compounds, and humus materials increase. Ammonia-producing, nitrifying, denitrifying, desulfurizing, and sulfurizing bacteria all play a part. The pH should be 6.0 to 6.5; adequate moisture is necessary, but direct sunlight is harmful.

Occurrence of a coarsely crystalline kaolin mineral in some South African fire clays. V. L. Bosazza. *Amer. Mineralogist*, **26**, 290–92 (1941).—The fire clays contain nodules up to 1 cm. in length which chemical analysis and microscopic examination proved to be kaolinite. Thin sections of the fire clay between crossed Nicols, but without the use of conoscopic conditions, showed large grains with an acute bisectrix figure. These dark bars are probably due to strain during desiccation of the clay. Wormlike lath-shaped particles are also fairly common. Replacement of quartz by kaolinite is frequent. W.D.F.

Paragenesis of the pegmatite minerals of Striegau, Silesia. Wilson D. Michell. *Amer. Mineralogist*, **26**, 262–75 (1941).—Pegmatites in a granite mass of Striegau contain many open druses, in which were deposited most of the minerals studied. Five stages of mineralization have been found: (1) the magmatic stage of the pegmatite, with the formation of graphic quartz and orthoclase deposits surrounding the druses; (2) the albitization stage, not separated by a depositional break, where albite replaced orthoclase and was also deposited separately; (3) the stage where, after a depositional break, small quantities of many minerals were deposited, viz., magnetite, sphalerite, biotite, chlorite, tourmaline, fluorite, muscovite, molybdenite, epidote, and axinite; (4) the zeolite stage, with heulandite, stilbite, laumontite, and chabazite; and (5) the calcite stage, with abundant deposition of calcite. W.D.F.

Petrofabric relations of nepheline and albite in litchfieldite from Blue Mountain, Ontario. H. W. Fairbairn. *Amer. Mineralogist*, **26**, 316–20 (1941).—Petrofabric analysis shows that (1) the tabular habit of the albite produces a foliation and its elongation produces a lineation; (2) (010) of albite is parallel to the foliation, and (3) the long dimensional axes of both albite and nepheline parallel each other and the lineation. The albite orientation is probably the result of magmatic flow, and the nepheline orientation is influenced by the albite. W.D.F.

Porcelain jasper of Dudweiler—its composition and relation to ceramic bodies. Kurt Obenauer. *Ber. Deut. Keram. Ges.*, **21** [9] 371–81 (1940).—The clay slate at Dudweiler, near Saarbrücken, was burned in the vein continuously for eons and changed to porcelain jasper, so that it acquired the characteristics of an artificial ceramic product. The composition of this burned product is similar to that of the original material, the clay slate being changed to dense, fine-grained layers rich in sillimanite which show melted quartz and new formations of andalusite. The sandy layers were sintered to porous products and show a clearly recognizable glass basis, melted quartz grains, and a red coloration due to later infiltration of solutions containing iron. The fact that the remains of plants are not modified, as well as the simultaneous occurrence of glassy components and bubble spaces, points to a softening and then a resolidification in place under the pressure of the overlying mountain mass. On comparison with artificial ceramic fragments, agreement is found only when the layers of the porcelain jasper rich in quartz are compared with whiteware. The pure clay slates have no similarity to typical porcelains in their composition because of the new formation of andalusite, the very rare occurrence of melted quartz, and the lack of transparency even when viewed through very thin edges. W.H.H.

Pressure filter for use in determining exchangeable bases in soils. Alexius Jung. *Bodenkunde & Pflanzenernähr.*, **17** [1–2] 109–11 (1940).—J. describes a glass pressure filter whose outlet permits heavy filterable pastes or precipitates to filter quantitatively in a short time and to be washed in the process. The method of estimating exchangeable bases in soils is well understood to depend upon displacement of cations by treatment of the soil with an aqueous solution of strong salts, such as ammonium chloride or acetate. The customary procedure, according to Schollenberger, has been to modify the soil by treating it with $N/1$ ammonium acetate solution, filtering it, and then treating it again with ammonia acetate until the soil alkalis and alkaline earths are lost; alcohol washes out the excess ammonium acetate, causing them to disappear. The alkali and alkaline earth in the filtrate are estimated in the residue by adsorption capacity. The specific disadvantages shown by experience to be inherent in the ammonium acetate or suction methods are loss of time, wastage of material, and corresponding unreliability of results. To eliminate such difficulties, the Berlin Institute for Soil Science has developed a vacuum-filter apparatus using exclusively filtration by hydraulic pressure. Its enclosed form makes possible agitation of the soil pastes at any time during the filtration process. The closing of the filter pores is hindered by a mechanical device, and the soil is so intensively mingled with the cleansing solution that the filtrate goes through unclouded. By vibration of the glass partition, the adhering soil portions are rinsed in preparation for further washing which is easily repeated in the filter layer. Suction-filter processes are bothersome and time-consuming in the necessary interchanges of filtrate contents; in the pressure filter these are simply abolished, filtration being carried through at one session. In addition, a quantitative flushing with the least mass is obtained in a given time and flow. Numerous researches show that the soil parts themselves, when of stiff, clayey earths, are difficult and tedious to filter. With the aid of a pressure filter, they go through quickly and are clear; with only a little flushing they can be quantitatively washed out. In the pressure filter, such soils require only 30 min. instead of many hours or even days with former methods. When unforeseen circumstances cause interruption of filtration by the ammonium acetate process, the danger of the soil drying out is encountered; with the pressure filter, the stopcocks are merely turned, the outflow is closed off, and the reagent glass is turned down. By combining several such pressure filters with one hydraulic pump, a greater number of research series can be carried on in the shortest time. The apparatus should not be confined to the soils labora-

tory alone but should be used everywhere when filtration of very difficult masses is under consideration. K.R.

Progressive metasomatism of serpentine in the Sierra Nevada of California. GORDON A. MACDONALD. *Amer. Mineralogist*, **26**, 276–87 (1941).—Near Humphreys, serpentine nodules in a mica schist have been altered by emanations from an adjacent quartz diorite. Cores of residual serpentine are surrounded by successive envelopes of talc, talc and actinolite, actinolite, chlorite, and biotite. Chemical and Rosiwal analyses of the zones in the nodules show that the changes involve loss of magnesia, addition of silica in the inner zone, and addition of small amounts of other oxides. This resembles granitization, but because of the extremely basic composition of the original nodule the product is different. The tendency has been to produce mineral phases in equilibrium with the crystallizing magma rather than a rock identical with it. W.D.F.

Quantitative determination of minerals in clays by the X-ray method. E. MAEGDEFRAU AND U. HOFMANN. *Ber. Deut. Keram. Ges.*, **21** [10] 383–412 (1940).—The quantitative determination of the mineral components of clays was accomplished by an X-ray method. The method was placed on the most simple basis possible in order to exclude all the principal sources of error. The photometer curve of the Debye-Scherrer exposure was compared with a photometer curve of a mixture of pure minerals. The method is especially suitable for the analysis of binary mixtures and is applied only to the crystalline part of the clay, which has been shown by experience, however, to be the major part and to have the greater significance. The following mixtures were worked out: (1) quartz and kaolinite, (2) quartz and montmorillonite, (3) quartz and a micaceous clay mineral, (4) kaolinite and a micaceous clay mineral, and (5) kaolinite and montmorillonite. The limit of error is especially low with mixtures containing a small amount of quartz. In such cases it may be as low as about 1%. The method was used to analyze some important kaolins and clays, among others a mixture of Wyoming bentonite, Hettenleidelheim clay, and Kärlich blue clay. W.H.H.

Quartz xenocrysts in olivine basalt from the southern Sierra Nevada of California. ROBERT W. WEBB. *Amer. Mineralogist*, **26**, 321–37 (1941).—Basalts in the Kernville area northeast of Bakersfield are normally composed of microphenocrysts of olivine, often altered to iddingsite, labradorite laths, and magnetite, in a ground mass. Corroded quartz grains are widely distributed; other inclusions are pegmatite and granodiorite. Mechanical contamination of the basalt by granodiorite and granite xenoliths, with limited formation of new minerals, is responsible for the quartz grains. This theory is substantiated by the following facts: (1) all stages of granodiorite alteration are present; (2) small inclusions are mainly quartz, but with all gradations to granodiorite; and (3) olivine and quartz have not completely reacted. The angularity of the quartz grains with the absence of crystal boundaries and the granular masses of quartz with many different optical orientations warrant the conclusion that no direct or indirect crystallization of quartz took place. W.D.F.

Radioactivity of rocks. R. D. EVANS AND CLARK GOODMAN. *Bull. Geol. Soc. Amer.*, **52** [4] 459–90 (1941).—Most measurements of radioactive earth materials when applied to geology and cosmology have had qualitative value only, because of inadequate analytical care. In this study, several hundred measurements based upon a systematic program of standardization, calibration, and interchecking were made on acidic, intermediate, and basic igneous rocks and sedimentary rocks. The present averages are substantially lower than those obtained prior to 1936, and they show a marked decrease of radioactivity with increasing basicity. The Th/U ratios are in better agreement with geochemical data. Holmes' widely used estimates of the rate of generation of radioactive heat within the earth may need to be substantially decreased. See "Age . . .," this issue, p. 177. A.C.B.

Rate of solution of crystals. R. H. WILHELM, L. H. CONKLIN, AND T. C. SAUER. *Ind. Eng. Chem.*, **33** [4] 453–57 (1941).—As a background for solution-agitation studies, an integration without limiting restrictions is presented for the diffusion-rate equation involved when particles of sodium chloride are freely suspended and dissolved in water. The integral is solved graphically and is presented as a family of curves involving three dimensionless variables. The following individual variables are taken into account: volume of water, amount of salt, size and shape of salt particles, concentration of solution, and increase in volume of solution as dissolution of the solid salt proceeds. Experimental evidence is presented to show that the derivation is valid over the entire concentration range of sodium chloride solutions. An effective diffusion film thickness of 0.016 mm. is calculated for specific agitation conditions. Illustrated. F.G.H.

Relation between the geology of the deposits and the ceramic characteristics of the kaolin and clay. H. SÄRCHINGER. *Ber. Deut. Keram. Ges.*, **21** [7–8] 293–310 (1940).—Ordinarily, the geologist seeks to know the order in which events occur; he is a historian. He should cooperate with the ceramist, however, in considering the utility of geological products. The geological history of a ceramic raw material determines its field of application. Particle size depends on the structure of the mother stone, as do also the shrinkage, green strength, and the plasticity, which is greatest for the pitchstone kaolin because of its fineness. A question often discussed is the manner and cause of formation of a kaolin deposit. Most deposits were transported by water, but some are of such thickness that they can be explained only by a landslide. The mother stone of Seilitz kaolin is a very dense variety of quartz porphyry which strongly resists kaolinization; therefore the kaolin contains considerable feldspar and can almost be considered as a substitute for feldspar. In general, the quality of a clay and the purpose for which it is used depend on the value of the original rock subjected to erosion. Most researches have been confined to kaolin derived from porphyry, but kaolins are also derived from granites and other stones. Although, chemically, quartz porphyry and granite are similar, their differences in structure affect the resulting kaolins. The presence of biotite in the mother rock causes contamination of the kaolin with iron. As it is not white firing, this kaolin must be used for such purposes as the preparation of refractories. S. considered materials used in heavy ceramics. Fourteen clays were tested in regard to properties important in the dry-press process. Fineness alone did not suffice to explain the plastic characteristics. From a study of the data of Lehmann and Endell on whiteware clays (see "Earthenware . . .," *Ceram. Abs.*, **15** [1] 38 (1936)), it was found that the maxima for plasticity, green strength, and shrinkage run parallel with the sorption capacity. The sorption capacity is related to the amount of sedimentation. Further tests are needed to obtain a clear picture of the effect of the structure of the mother rock and the fineness of the clay upon the characteristics of the clay. W.H.H.

Relations between the most important characteristics of fired brick and tile, determined both in the laboratory and in operation. O. KALLAUNER. *Ber. Deut. Keram. Ges.*, **21** [9] 315–29 (1940).—Shrinkage on drying, with the same consistency of the molded piece, was greater for small pieces than for normally large pieces. This confirmed Wilson's contention that test results on small pieces are generally not applicable to larger pieces. No great difference could be determined in the characteristics of small test pieces of different form fired in the laboratory to Seger cone 010*a*. At cone 07*a* and higher temperatures, however, distinct differences could be observed; e.g., with decreasing size, absorption and compressive strength increased. Pieces fired for 4 hr. in the laboratory to cone 010*a* showed no great difference in characteristics from those fired for 9 hr. Variations in the time of firing, however, had a noticeable effect on the values of absorption capacity when normal brick were compared with those fired in a circular brick kiln or in a

laboratory gas kiln. For normal brick with a firing period of 9 hr. in an oxidizing atmosphere, it was necessary to fire about 3 cones higher in the laboratory to obtain characteristics (mainly in regard to absorption) similar to those of brick fired in a circular brick kiln. For normal roofing tile and small laboratory articles, the difference was smaller (about 2 cones). The smallest differences were found with clinker clays containing iron and poor in lime; these differences did not become apparent until cone 05*a*. Values of absorption (n_v) and compressive strength (p) confirm the approximate empirical equation relating these important properties: p kgm./cm.$^2 = \frac{112{,}500}{n_v^3 + 50}$. In spite of the greatest care, test results may be affected by (1) noticeable differences in the petrographic composition and dispersion or differences in the consistency of the bodies, (2) temperature differences in different parts of the kiln, and (3) fluctuations in the atmosphere of the tile kiln. Further experiences indicate that the empirical rules for judging tile clay in the laboratory which resulted from this study are also valid for the great number of brick earths. In some cases, differences which occur can be explained as the result of special circumstances. W.H.H.

Spectrographic study of Central American and Asiatic jades. D. Norman and W. W. A. Johnson. *Jour. Optical Soc. Amer.*, **31**, 85–86 (1941).—Jades from Yucatan, China, Burma, New Zealand, and New York were analyzed spectrographically. No marked chemical difference exists between the American and oriental jades, but the American specimens contain a larger proportion of diopside. A.P.

Statistics of the ceramic industry in Canada. W. H. Losee. *Jour. Can. Ceram. Soc.*, **9**, 27–39 (1940).—For convenience, clay products manufactured in Canada are classified, for statistical purposes, into (1) those made from domestic clays and (2) those made from imported clays. Statistics are given on the following classes of ceramic products manufactured in Canada: artificial abrasives, cement, glass, and sand-lime brick. J.G.P.

Stress-strain hysteresis in certain clays and other substances. H. H. Macey. *Nature*, **147** [3718] 146–47 (1941).—Certain clays prepared commercially by sedimentation, when held in the fingers of both hands and alternately pulled and compressed, have a range of easy movement culminating in a sudden resistance. A stress-strain hysteresis loop of such material between equal positive and negative values shows appreciable movement at low stresses. M. calls the property "backlash." China clays behave in this manner to a small degree. M. suggests that backlash is due to a sliding of the flat particles relative to one another. In the majority of clays which do not show this effect, the presence of finer particles in the interstices between the larger particles prevents this movement. 2 hysteresis curves. J.L.G.

BOOKS AND SEPARATE PUBLICATIONS

Chrome Ore and Chromium. Reports on the Mineral Industry of the British Empire and Foreign Countries. Robert Allen and G. E. Howling. Imperial Institute, Mineral Resources Department, London, 1940. 118 pp. Price 2s 6d net, postage 4d.—During the period between 1918 and 1939, the output of chrome ore increased from less than a quarter of a million tons a year to more than a million. This monograph presents facts concerning chrome ore and chromium as they were at the outbreak of the war in September, 1939. There is a brief discussion of the ore and its mining, dressing, and uses, followed by reference to marketing, prices, and world production, with accompanying tables of statistics. Chromium is used for many nonferrous alloys, e.g., Stellite, which is very essential for machine tools. The demand for improved refractory linings for furnaces in steel production is being met to an increasing extent by the use not only of chrome brick but more especially by composite refractories, such as chrome-magnesite brick, which possess greater refractoriness under load and improved resistance to spalling. The main portion of the monograph gives details on the occurrence of chrome ore throughout the world. About 43 countries are included, with data on location of deposits, grade of ore (with tables of analysis in some cases), and imports and exports. A lengthy list of literature references is included. The book is of value for everyone concerned with the production and use of chrome ore and is of interest to economists, teachers, and the general reader. F.G.H.

Elementary Crystallography. J. W. Evans and G. M. Davies. Nordeman Publishing Co., Inc., New York, 1940. vii + 149 pp., 203 illustrations. Price $2.00. Reviewed in *Amer. Jour. Sci.*, **239** [5] 393 (1941).—This is a small, compact treatise on the fundamentals of crystallography. B.C.R.

Indian Refractory Clays. Anon. *Bull. Ind. Research Bur., Govt. of India*, No. 14, 63 pp. (1939).—Indian refractory clays are of three kinds: clays of sedimentary origin, kaolins formed as a result of the decay of feldspars in granitic rocks, and lithomarges which are formed below the surface of the ground as a result of the disintegration of surface rocks. The author has collected and collated a large amount of information about clay deposits in India hitherto scattered throughout various official files and publications, chiefly those of the Geological Survey of India. Each kind of clay is described in a separate chapter. The main classification of the clays has been arranged geographically so that information about the clays available in any locality is readily ascertainable. The appendix contains analyses of a large number of Indian fire clays and Indian and foreign firebrick, valuable statistical information, and a bibliography. A good map is included. A.P.S.

Outlines of Structural Geology. E. Sherbon Hills. Nordeman Publishing Co., Inc., New York, 1940. ix + 172 pp., 2 plates, 105 figs. Price $2.25. Reviewed in *Amer. Jour. Sci.*, **239** [5] 393 (1941).—A brief but reasonably complete summary of structural geology is presented with special reference to those aspects of the subject with which the field geologist should be acquainted. B.C.R.

Southeastern Missouri Clays. W. E. Davis, P. G. Herold, and Lyle McManamy. *Missouri Geol. Survey & Water Resources, 61st Biennial Rept.*, Appendix I, 48 pp. (1941). 3 maps, 6 tables.—The report describes the occurrence and properties of clays contained in undeveloped deposits in Dunklin, Stoddard, and Scott counties in southeastern Missouri. Results are given of an extensive field and laboratory investigation conducted in cooperation with the WPA to determine the suitability of these clays for the manufacture of ceramic products. Many of the deposits listed contain clays which are quite suitable for the manufacture of pottery and stoneware. Some of these clays may be of sufficiently good quality after washing to permit their use as a substitute for ball clay in whiteware bodies. Others are suitable for the manufacture of terra cotta, brick, tile, retorts, and lightweight aggregate. It is believed that a few of the clays reported may be used as plasticizers in cement mortars. Tables give the physical properties, drying and firing behavior, fusion points, chemical analyses, and possible commercial uses of the clays tested. Logs of holes drilled during the prospecting of deposits are also given. Copies of the report may be obtained from H. A. Buehler, State Geologist, Missouri Geological Survey, Rolla, Mo. A small fee will be charged to cover printing and mailing costs.

PATENTS

Purification of silica sand. R. T. Hancock. Brit. 535,455, April 23, 1941 (Feb. 15, 1940).

Treating clay. P. D. Barton (Sun Oil Co.). U. S. 2,242,639, May 20, 1941 (March 29, 1938).—The method of treating clay containing adsorbed oil to remove oil therefrom.

Treatment of colloidal clay. Sin Iwamura. Japan. 132,027, Sept. 7, 1939; *Chem. Abs.*, **35**, 3053 (1941).—Clay containing colloidal substances and water is treated with dilute mineral acid, and an alternating current is

applied in order to facilitate filtration to separate the clay from the water.

Treatment of silica ores. G. C. WESTBY (Soluble Silicates Process Co., Inc.). U. S. 2,243,044, May 20, 1941 (April 17, 1937).—The process of treating manganiferous silicate ores to obtain the manganese in concentrated form comprises sufficiently heating such an ore in a pulverulent state with an excess of caustic soda, while maintaining the ore in an oxidizing atmosphere, to produce a fused mass of sodium hydrate, sodium manganate, sodium permanganate, sodium silicate, sodium ferrate, and a suspended unacted-on residue, forming and discharging a molten stream from the fused mass, shredding the stream in the presence of water to disintegrate the stream and leave the undissolved residue while taking the soluble components of the stream into solution, and treating the resulting hot alkaline solution of sodium manganate, sodium permanganate, sodium silicate, and sodium hydrate with a carbonaceous substance to precipitate hydrated manganese dioxide.

Chemistry and Physics

Alumina floc. HARRY B. WEISER, W. O. MILLIGAN, AND W. R. PURCELL. *Ind. Eng. Chem.*, **33** [5] 669–72 (1941).—The composition of alumina floc formed at pH values below 5.5 is described in detail. Illustrated. See *Ceram. Abs.*, **20** [2] 56 (1941). F.G.H.

Coagulation of magnesium fumes. J. BRICARD. *Compt. Rend.*, **211** [14] 278–80 (1940).—When Mg powder is burned and the fumes are condensed on a small glass plate, small crystals are formed (about 0.3 micron); at first they are separate, but in time they unite. Their density changes from 0.258 to about 0.62, averaging about 0.309. The number of particles formed in the unit of volume can be represented by the Whytlaw-Gray formula. M.H.

Colloid chemistry in ceramics. E. A. HAUSER. *Jour. Amer. Ceram. Soc.*, **24** [6] 179–89 (1941).—15 figures.

Determination of antimony in antimony ores by the potassium bromate method. L. CUDROFF. *Chemist-Analyst*, **30** [1] 4–6 (1941).—Detailed precautions are given for obtaining accurate results. A.P.

Determination and separation of potassium as periodate. HOBART H. WILLARD AND ALBERT J. BOYLE. *Ind. Eng. Chem., Anal. Ed.*, **13** [3] 137–39 (1941).—Potassium can be quantitatively precipitated as periodate by adding periodic acid to a solution only a few milliliters in volume and subsequently completing the precipitation by the addition of a much larger volume of a mixture of equal parts of aldehyde-free ethyl alcohol and anhydrous ethyl acetate. The solution must be maintained at 0°C. and stirred for 30 min. The precipitate of potassium periodate may be weighed, or it may be dissolved in a boric acid-borax buffer, potassium iodide added, and the free iodine titrated with arsenite. In this reaction the periodate is reduced to iodate. Potassium may be separated from calcium, magnesium, zinc, aluminum, sodium, lithium, nickel, and cobalt but not from manganese, iron, chromium, rubidium, cesium, and ammonium. It is possible to separate as little as 0.4 mgm. of potassium from seventy times as much sodium. If both calcium and sulfate are present the results are too low, probably because of the formation of a double potassium calcium sulfate. Small amounts of sulfuric, phosphoric, nitric, and boric acids may be present, but with large amounts the precipitate becomes gelatinous and difficult to filter. In the presence of sulfate, the gravimetric determination is impossible, but the volumetric method gives satisfactory results. Chloride must be absent. The method is rapid and accurate. F.G.H.

Determination of zinc by precipitation as zinc anthranilate. C. W. ANDERSON. *Ind. Eng. Chem., Anal. Ed.*, **13** [5] 367–68 (1941).—A gravimetric semimicromethod is described. F.G.H.

Dielectric properties of the rutile form of TiO_2. L. J. BERBERICH AND M. E. BELL. *Jour. Applied Physics*, **11**, 681–92 (1940).—Three specimens of rutile were prepared (1) at 1675° for 2 hr., (2) at 1330° for 6 hr., and (3) with 5% ceramic binder. Power-factor and dielectric-constant data were measured over a range of frequency and temperature. A dielectric constant of about 100 with low dielectric losses over a wide frequency range was found. The dielectric constant decreases with increasing temperature, having an average temperature coefficient of -8.2×10^{-4} per degree. An exponential relationship between conductivity and $1/T$ holds in the higher temperature region. Mixtures of rutile powder with other dielectric materials obey a logarithmic mixture formula. Two mechanisms for the high dielectric constant are discussed. A mechanism based on the Clausius-Mosotti equation offers a plausible explanation. A.P.

Metallic reflection by compressed crystalline powders. J. A. SANDERSON. *Jour. Optical Soc. Amer.*, **30**, 566–67 (1940).—Crystalline powders, when pressed into plates at 40,000 lb. per in.^{2}, reflect strongly in the infrared spectrum at wave lengths characteristic of their functional groups. Such plates may be useful in optical investigations of materials not available as large crystals. Reflection curves for the region 6 to 13 microns are given for finely divided powders of calcium carbonate, calcium sulfate, plaster of Paris, gypsum, talc, silver nitrate, and potassium dichromate. A.P.

Method of determining nickel and cobalt in meteoric iron. E. P. HENDERSON. *Amer. Jour. Sci.*, **239** [5] 372 (1941).—H. presents an analytical procedure for the determination of nickel and cobalt in the presence of considerable amounts of iron in which the nickel may be separated by precipitation of the iron with a large excess of ammonium hydroxide. Results indicate that the nickel will precipitate with the iron if only a slight excess of ammonia is used, a factor which produced errors in earlier analyses in which the iron was determined gravimetrically. B.C.R.

Microanalysis of gases by physical methods. C. E. RAMSLEY. *G.E.C. Jour.* [London], **11**, 135–41 (1940).—R. describes in detail the accurate analysis of mixtures of gases occupying as little as 0.001 ml. at standard temperature and pressure by the low-pressure method first introduced by I. Langmuir. The constituent gases are identified and measured by a succession of chemical or physical processes which are recorded by McLeod or Pirani gauges. Condensable gases such as SO_2, CO_2, and H_2O may be successively condensed, or the three may be condensed with liquid air and warmed up when each exerts its vapor pressure in turn. The permanent gases are dealt with separately. H_2 is obtained by diffusion through a palladium tube at 700°C. The CO left is burned to CO_2 with O_2 from the air (admitted selectively from the air through a heated silver tube); the CO_2 is then frozen out. The N_2 is absorbed on a tantalum filament at 1200°C. The method is suitable for CO and CO_2 in flue gases for which separate samples of 0.03 to 1 ml. may be used. Tests of the method show good agreement with Hahn's modified Orsat apparatus. L.R.B.

pH-measurements with new glass electrodes of high-conductive glass. K. SCHWABE AND O. HERDEY, JR. *Zellstoff & Papier*, **19**, 530–33, 567–70 (1939); abstracted in *Physik. Ber.*, **21** [7] 702 (1940).—New type glass electrodes are made of Schott glass A638/84 141, unsilvered, and 9000/86 035, silvered; they were compared with an electrode of MacInnes glass. It was found that the shifting of the potential with the change in pH corresponds less exactly to the Nernst formula in the new electrodes than in a glass with 72 SiO_2, 6 CaO, and 22% Na_2O; pH shifting caused by the giving off of alkali is stronger in the new glasses than in the MacInnes glass and cannot be compensated for as is possible in the latter glass. The new glasses also show temporary variations in potential. M.H.

Physical basis for spectrographic analysis. W. F. MEGGERS. *Jour. Optical Soc. Amer.*, **31**, 39–46 (1941).—Scientific answers to questions puzzling spectrochemical analysts are to be found in the optical properties of spectrographs, the physical characteristics of light sources, and the mechanism of atomic radiation. Electric arcs are best for exciting the first spectra of the elements. The strongest lines result from a transition between *s* and *p* electrons in the atoms. If the normal state contains a single *s* electron, the atoms have extraordinary spectral sensitivity. Valence electrons of the *p* type are more firmly bound; noble gases, halogens, and metalloids, therefore, have low sensitivity. The success of practical spectrographic analysis for seventy elements is related to the fact that these elements have low ionization potentials and strongest lines above 2000 a.u. A.P.

Precipitation of aluminum as hydroxide. E. W. KOENIG. *Chemist-Analyst*, **30** [1] 15 (1941).—Bromcresol purple is recommended in place of methyl red to indicate the end point in ammonium hydroxide precipitation of aluminum. A.P.

Qualitative spectrochemical analysis in agriculture and geochemistry. S. S. BALLARD. *Jour. Applied Physics*, **11**, 750–58 (1940).—Qualitative spectrochemical methods are useful for determining the approximate composition of a totally unknown sample and for identifying and estimating the amounts of minor and trace elements. In rocks, barium, chromium, zirconium, vanadium, strontium, nickel, copper, etc., lie within the accurate range of the spectrograph. Precipitates can be tested for traces of impurities. Rocks can be checked to see whether trace elements are uniformly distributed or are concentrated in certain mineral constituents. A.P.

Quantitative determination and separation of copper with benzotriazole. J. ALFRED CURTIS. *Ind. Eng. Chem., Anal. Ed.*, **13** [5] 349–51 (1941).—A new organic precipitant for copper, benzotriazole, is described. Copper is precipitated in a tartaric-acetic acid solution at a pH of 7.0 to 8.5. In the absence of silver, nickel, cadmium, zinc, cobalt, and ferrous iron, the precipitate can be weighed directly. In their presence the reagent can be used to effect a preliminary separation, and the copper can be subsequently determined by a standard method, e.g., electrolytically or by the potassium iodide-thiosulfate method. F.G.H.

Rapid and accurate determination of cadmium. T. L. THOMPSON. *Ind. Eng. Chem., Anal. Ed.*, **13** [3] 164–65 (1941).—Standard procedures call for the separation of zinc and cadmium by repeated precipitations with hydrogen sulfide or by electrolysis under carefully controlled conditions of pH and current density involving the use of various addition agents. Because of the consanguinity of zinc and cadmium, these methods are not satisfactory. In 1937 Korenman suggested the precipitation of cadmium from a cadmium-zinc solution by a brucine-potassium bromide mixture, followed by its filtration, washing, drying, and weighing. The objectionable features of the method were the long period of precipitation, the impossibility of washing clean, and the tendency of the precipitate (due to its great bulk) to decompose, volatilizing the bromine, before completely drying. Its advantages were the completeness of separation and the cheapness and cleanliness of the chemical. Experiments have shown that if the cadmium is precipitated from the cadmium-zinc solution with a brucine sulfate-potassium iodide mixture and then washed by special means, the precipitate can be dissolved, and the cadmium can be determined by titrating the iodine present; this method gives accurate and rapid results if carefully carried out. F.G.H.

Reaction in the solid state between SiO_2 and CaO, kaolin, its firing products, and CaO. P. P. BUDNIKOV AND D. P. BOBROVNIK. *Trudy Mineralogii & Petrografii (Trans. Mineralogy & Petrography, Inst. Geol. Sciences, Acad. Sciences U.S.S.R.)*, **1940**, 223–29.—The formation of calcium silicates from CaO and SiO_2 at 500°, 600°, and 700° was investigated. With the increase in temperature, the amount of CaO reacting with SiO_2 increases, and with increase of grain size, it decreases. In samples fired at 1000°C., water-soluble and insoluble calcium silicates were formed, the amount of free CaO decreasing compared with that of samples fired at a lower temperature. At 600° more α-calcium orthosilicate (C_2S) and less C_3S_2 and CS form; at 1000° the formation of CS and C_3S_2 predominates, and only an inconsiderable amount of α-C_2S is found. It follows that the reaction in the solid phase proceeds according to the Ostwald law by leaps over intermediary unstable compounds ($2CaO \cdot SiO_2$ and $3CaO \cdot 2SiO_2$) to the final $CaSiO_3$. The rate of reaction increases with the temperature and with increase of activity of SiO_2 and degree of dispersion of CaO. These findings have been applied to the firing of silica brick. P.B. & E.S.

BOOKS

Bibliography of Spectrochemical Analysis. Compiled by D. M. SMITH. British Non-Ferrous Metals Research Assn., London, 1940. 2d ed. 55 pp. Price 3s. Reviewed in *News Ed.* (Amer. Chem. Soc.), **19** [3] 166 (1941); see "Quantitative . . ." *Ceram. Abs.*, **20** [4] 100 (1941). F.G.H.

Physical Chemistry of Silicates (Physikalische Chemie der Silikate). WILHELM EITEL. Johann Ambrosius Barth, Leipzig, 1941. 2d ed. 826 pp., 653 illustrations. Price Rm. 54.—This entirely revised and enlarged edition contains much new material pertaining to the chemistry of silicates. Chapters deal with the colloidal, crystalline, glassy, and liquid states of silicates, the behavior of the clay–water system, and the physical chemistry of hydraulic cements. The great progress in researches in glass is amply represented. See *Ceram. Abs.*, **8** [10] 773 (1929). L.E.T.

Raman Effect and Its Chemical Applications. JAMES H. HIBBEN. American Chemical Society Monograph Series, No. 80. Reinhold Publishing Corp., New York, 1939. 544 pp. Price $11.00.—The book consists of three parts: I, A General Discussion of the Raman Effect, Its Practice, and Its Theory; II, The Raman Spectra of Organic Compounds; and III, The Raman Spectra of Inorganic Compounds. A comprehensive bibliography and an elaborate index are given. An excellent account of the available experimental methods is given. The theoretical aspects of the subject, with special reference to the normal vibrations of polyatomic molecules, are dealt with in an elementary manner. The mathematical detail has been omitted in a number of places as its presentation would have been out of place and would have unduly increased the size of the book. In Parts II and III, the subject of molecular constitution is the dominating feature. Several other aspects such as isomerism of different kinds, free rotation, electrolysis, polymerization, etc., are adequately dealt with. A discussion of the Raman spectrum of benzene in relation to its constitution is presented. A full description of the Raman spectra of several simple substances, such as some gases, phosphorus, sulfur, carbon, water, inorganic acids, etc., is given which forms the basis for a thorough understanding and appreciation of the various constitutional problems. An up-to-date and exhaustive collection of experimental results so far obtained on the subject of Raman spectra is included. The book will be of great help as a reference for all research workers in this subject. A.P.S.

PATENTS

Chlorination of titanium-bearing materials. PITTSBURGH PLATE GLASS CO. Brit. 535,178 and 535,179, April 17, 1941 (June 30, 1939).

Colored pigment. C. J. HARBERT (Harshaw Chemical Co.). U. S. 2,243,033, May 20, 1941 (Jan. 12, 1939).—A pigment material, being a calcination product of a manganese oxide yielding material other than a manganese salt of a strong mineral acid, aluminum hydrate, and a minor quantity of calcium fluoride, manganese oxide being present to the extent of 5 to 20% of the aluminum oxide.

Hydrous silicate gels and method of making. E. W. REMBERT (Johns-Manville Corp.). U. S. 2,241,791, May 13, 1941 (July 7, 1939).

Manufacture of anhydrous silicates in pumiceous form. C. L. BAKER (Philadelphia Quartz Co.). U. S. 2,243,027, May 20, 1941 (Aug. 24, 1938).—A pumiceous, anhydrous sodium silicate in the form of a congealed anhydrous melt, when in bulk resembling in appearance a fine-grained bread with minute bubbles dispersed in a matrix of fine crystals and vitrified material, the product containing about 2 to 0.5 mol. of SiO_2 to 1 mol. of Na_2O, having a specific gravity ranging from about 0.5 to 2.5, and having a rate of solubility of the order of that of a solid hydrated product having a similar granulation and ratio of SiO_2 to Na_2O, the bubbles in the product resulting from the internal evolution of a gas in a melt of the product during cooling and crystallization and before hardening.

Manufacture of cement and alkali-metal aluminate. WALTER NOLL (I. G. Farbenind. A.-G.). U. S. 2,242,258, May 20, 1941 (Jan. 12, 1939).

Manufacture of silicates. D. B. CURLL, JR. (Philadelphia Quartz Co.). U. S. 2,239,880, April 29, 1941 (Jan. 14, 1937).—(4) In the production of alkaline-metal silicates, the process comprises mixing together silica sand with an alkaline-metal salt having a melting point which is lower than the melting point of the sand and lower than that of the silicate product to be produced in molecular proportions within the range of about $2SiO_2$:1 alkaline-metal oxide to $1SiO_2$:2 alkaline-metal oxide, the alkaline-metal salt having an acid radical which is capable of being volatilized during the course of the reaction, thereby raising the temperature at which the reaction mixture will form a melt, heating the resulting mixture under conditions producing a uniform distribution of temperature through the mixture, the substantially complete elimination of the acid radical, and the formation of an alkaline-metal silicate, the heating temperature at the start of the process being maintained below the fusion temperature of the alkaline-metal salt until a substantial portion of the acid radical has been volatilized, the heating temperature then being raised above the fusion temperature to complete the reaction but being maintained throughout the process below temperatures producing any substantial fusion of any of the ingredients of the reaction mixture, whereby a porous silicate product of nonvitreous appearance is produced. (12) As new compositions of matter, porous calcination products having a nonvitreous appearance comprising an alkali-metal silicate having a molecular ratio of SiO_2 to alkali-metal oxide ranging from about 2:1 to 1:2, the products containing finely divided, interlocked crystals of an anhydrous alkali-metal silicate, giving the product an opaque appearance, the larger granules being oölitic in appearance, the granules consisting largely of crystals of sodium metasilicate in the form of dendrites radiating from common centers like the seeds of a cocklebur, the common centers consisting of a silicate glass, the more siliceous of the products also containing crystals of alkali-metal disilicate, the products being readily and substantially completely soluble in water and having an apparent density ranging from about 0.6 to 1 when sufficiently fine to pass a 65-mesh screen.

Preparation of titanium dioxide. F. J. CLEVELAND (Pittsburgh Plate Glass Co.). Brit. 535,214, April 17, 1941 (Aug. 29, 1939). PITTSBURGH PLATE GLASS CO. Brit. 535,213, April 17, 1941 (May 4, 1939).

Production of beryllium compounds. HELMUT VON ZEPPELIN (W. H. Duisberg). U. S. 2,242,492 and 2,242,493, May 20, 1941 (April 1, 1939).

Removing silica from minerals, slags, etc. EDUARD ZINTL, WALTER KRINGS, AND WILHELM BRÄUNING (W. H. Duisberg). U. S. 2,242,497, May 20, 1941 (April 3, 1939).—A process of removing silica from minerals, slags, and similar materials containing difficultly volatilizable metal oxides chemically combined as silicates, the metal oxides being difficultly reducible with silicon, comprises adding silicon to such materials, heating the mixture of materials and silicon to temperatures above 1250°C. under a vacuum, thereby decomposing the silicate in the material with the formation of silicon monoxide, removing the silicon monoxide from the material as a gas, and leaving the oxides as a residue.

General

Capillary flow in porous solids. N. H. CEAGLSKE AND F. C. KIESLING. *Trans. Amer. Inst. Chem. Engrs.*, **36**, 211–25 (1940).—The authors define two types of flow: saturated flow exists when a fluid is forced through a porous media by means of an external pressure; capillary flow exists when a liquid is flowing through a porous media without the assistance of an external force. The force causing capillary flow is produced by the surface tension at the air-water interfaces and is called suction. The flow obeys a modification of Darcy's law, and the permeabilities of both saturated flow and capillary flow are alike until the total suction reaches the entry value. When the total suction exceeds the entry value, the capillary permeability rapidly drops to zero. H.E.S.

Ceramic industry in Switzerland. MARIE WIDMER. *Design*, **42** [5] 10–11 (1941).—W. gives a brief résumé of the history of the ceramic industry in Switzerland. The industry includes the following main branches: (1) tile, Dutch brick, and fire-clay products; (2) pottery and slabs for stoves; (3) glazed ware; (4) stoneware; and (5) china. The first and fourth groups manufacture almost exclusively for building and engineering purposes, although most of the firms concentrate on tableware and crockery. The only Swiss porcelain factory is at Langenthal. To give proper training to workers and future foremen and managers in the ceramic industry, the Swiss Ceramics School was founded at Chavannes-Renens near Lausanne in 1913, with the help of the Federal and Cantonal authorities. Illustrated. L.S.

Chemical resistance of sulfur cements. C. R. PAYNE AND W. W. DUECKER. *Trans. Amer. Inst. Chem. Engrs.*, **36**, 91–111 (1940).—Specific data are given regarding the resistance of sulfur cements to water; hydrochloric, nitric, sulfuric, phosphoric, and hydrofluoric acids; alkaline cleaners; oxidizing agents such as potassium dichromate; and various organic substances such as sugar, cream, soya-bean oil, etc. Typical structural adaptations are pointed out and illustrated. H.E.S.

Clay products in 1941. J. M. LANGE. *Ind. Marketing*, **26** [1] 46 (1941).—The refractory industry is expecting capacity production due to defense orders. The general trends in the ceramic industry are as follows: (1) more mechanical handling, (2) modern types of tractor digging equipment, (3) individual motor drives, (4) the use of stokers and natural gas for firing kilns, (5) improved drying operations, (6) more precision equipment and control devices, (7) greater use of Diesel and gas engines, (8) use of small-sized tunnel kilns, and (9) more mechanization in the sewer-pipe industry. F.E.S.

Comparison of methods for sampling lead fume. LEWIS B. CASE. *Amer. Jour. Pub. Health*, **31** [4] 359 (1941).—In tests conducted under controlled conditions, the efficiencies of impingers compared with those of electrostatic samplers are higher when dealing with relatively large concentrations, although no definite ratio could be established from the data so obtained. Comparisons made with asbestos filters indicated less inconsistency than that found with the impingers. Quantitative determinations of small quantities of lead fume are beset with various uncertainties and complications and should be interpreted accordingly. B.C.R.

Determination of fusion characteristics of nonplastic materials. ARTHUR J. BLUME. *Bull. Amer. Ceram. Soc.*, **20** [5] 153–54 (1941).

Drilling-dust problems. G. HILDICK SMITH. *Jour. Chem. Met. Mining Soc. S. Africa*, **40** [12] 382–407 (1940).

—A standard type of long-piston jackhammer was modified by adapting to it a front-head water feed, thereby greatly reducing the amount of air expelled from the hole and consequently the amount of dust carried out by the air. L.W.V.

Dust hazards. L. M. MERRITT. *Safety Eng.*, **81** [2] 35–36, 38; [3] 37–38 (1941).—The hazardous dust-producing operations in the whiteware industry are discussed. W.J.L.

Charles W. Franzheim. ANON. *Bull. Amer. Ceram. Soc.*, **20** [5] 185–86 (1941).

Industrial illumination: I–IV. D. M. WARREN AND A. R. FINLEY. *Steel*, **106** [Jan. 22] 36–40, 52–53; [Jan. 29] 51–56, 66; [Feb. 5] 49–53, 75; [Feb. 12] 57–61 (1940).—The characteristics of new light sources and new and improved fixtures are described, together with their application to industrial uses. Illustrated. H.E.S.

Measuring smokes and rating efficiencies of industrial air filters. A. C. ROBERTSON, J. G. MULDER, AND F. G. VANSAUN. *Ind. Eng. Chem., Anal. Ed.*, **13** [5] 331–34 (1941).—To attain the extremely clean air required by some industrial processes, the ultimate in air filtration is necessary. A technique is described whereby it is possible to classify air filters critically by continuous measurement of concentrations of smokelike materials in filtered and unfiltered air. By determining the density differences of impingement traces, it is possible to calculate the percentage efficiencies of air filters. The apparatus consists of an impingement recorder and a photoelectric densitometer. The recorder operates continuously by impingement of air from a self-metering orifice on moving paper. The unit is readily portable. Illustrated. F.G.H.

Outstanding developments in ceramics since 1936. G. A. KIRKENDALE. *Jour. Can. Ceram. Soc.*, **9**, 14–20 (1940).—K. reviews the technical and industrial developments in ceramics during the past four years. J.G.P.

Patent Committee suggestions. FULTON B. FLICK, *Chairman. Bull. Amer. Ceram. Soc.*, **20** [5] 174–75 (1941).

President's address. JOHN L. CARRUTHERS. *Bull. Amer. Ceram. Soc.*, **20** [5] 164–66 (1941).

Recognizing and combating dust in the ceramic industry. GEORG HASS. *Ber. Deut. Keram. Ges.*, **21** [10] 416–24 (1940).—The firm of Carl Zeiss, in Jena, has developed an instrument, called a "konimeter," for measuring the dust content of the air. The air is forced through an orifice and strikes a glass plate at high speed. The dust, retained on the plate by an adhesive, is examined with a microscope. This instrument furnishes the possibility of measuring the dust in workrooms and of determining the effectiveness of dust-removal apparatus. In discussing the portable dust remover and dust masks, H. points out recent experiments which could be applied in the ceramic industry. If the management and workers, without prejudice, apply in their own plants what has been learned elsewhere, it will be possible to gradually eliminate dust in plants. W.H.H.

Report of Committee on Research for 1940–1941. A. A. WELLS, *Chairman. Bull. Amer. Ceram. Soc.*, **20** [5] 166–74 (1941).—24 references.

Review of 1940. H. HALLIDAY. *Claycraft*, **14** [5] 131–35 (1941).—H. reviews the heavy clay industry in England for 1940 under the following headings: (*a*) scarcity of labor due to competition from other industries, (*b*) priorities and difficulties of transportation and fuel supplies, (*c*) refractories industry, (*d*) wages and labor questions, (*e*) obscuration of kiln glare, and (*f*) clay industries export group. G.A.K.

Silicosis in the ceramic industry. J. G. CUNNINGHAM. *Jour. Can. Ceram. Soc.*, **9**, 4–8 (1940).—Silicosis is produced in certain branches of the ceramic industry. Much can be done to control the hazard without encountering difficult and expensive engineering problems such as those confronting the mining industry. Silica occurring in nature in the combined form as silicates is not known to produce any important degree of lung fibrosis except in the case of asbestos and talc. Frit manufactured for use in vitreous enamel and amorphous silica in the form of diatomaceous earth or tripoli are regarded, as yet, with suspicion. The presence of accompanying minerals in a silica exposure, as is constant in ceramics, on theoretical grounds, may act as an inhibitor to the amount of silica inhaled or to its effect in the lung, but the number of cases of silicosis which develop in certain sections of this industry at moderate concentrations of dust does not suggest that any protective function of practical importance is exercised by those materials normally present. In common with other industries affected, physical examination on employment and periodically afterward is essential for the control of tuberculosis and the evaluation of measures in use for minimizing dust exposure. J.G.P.

James MacMath Smith, President of Shenango Pottery Co. ANON. *Bull. Amer. Ceram. Soc.*, **20** [5] 163–64 (1941).

Walter Steger's 25th anniversary of service. R. RIEKE. *Ber. Deut. Keram. Ges.*, **21** [11] 465–71 (1940).—Dr. Steger was born July 6, 1889. On Nov. 30, 1940, he finished 25 years of service with the testing laboratory of the Federal Porcelain Factory in Berlin. The list of his 94 published articles indicates his activity in all branches of ceramics. He has also written one book, translated another, and cooperated on three more. W.H.H.

Summary of some undergraduate theses from the New York State College of Ceramics. C. R. AMBERG. *Bull. Amer. Ceram. Soc.*, **20** [5] 158–62 (1941).—2 figures.

SEPARATE PUBLICATIONS

Italian Glass, Ceramics, and Refractories at the Autumn Fair in Leipzig, 1940 (Italienisches Glas, Keramik und feuerfeste Stoffe auf der Leipziger Herbstmesse, 1940). Published by Il Vetro and La Ceramica. Reviewed in *Ber. Deut. Keram. Ges.*, **21** [9] 381 (1940).—With the most thorough use of raw materials obtained in Italy, almost every kind of ceramic and glass object is made by modern methods. W.H.H.

Proceedings of Fifth Annual Meeting, Air Hygiene Foundation of America, Inc. Air Hygiene Foundation of America, Inc., Pittsburgh, 1940. 106 pp.—The following papers are included: **Report of the Preventive Engineering Committee.** PHILIP DRINKER, *Chairman.* Pp. 9–16. **Report of the Medical Committee.** A. J. LANZA, *Chairman.* Pp. 17–19. **Foundation study of sick absenteeism in industry.** W. M. GAFAFER. Pp. 20–26. **Individual susceptibility to toxic dusts.** L. U. GARDNER. Pp. 26–35. **Further developments of the observation of the reaction of living tissue to silica granules in the living mammal.** ELIOT R. CLARK AND DARROW E. HAAGENSEN. Pp. 36–41. **Review of recent occupational disease legislation.** THEODORE C. WATERS. Pp. 42–51. See "Sick...," *Ceram. Abs.*, **20** [4] 108 (1941).

Proceedings of the Industrial Hygiene Sessions, National Battery Manufacturers Assn., Chicago, October, 1939. National Battery Manufacturers Assn., Akron, Ohio. Price $2.00. Reviewed in *Ind. Med.* [Ind. Hyg. Sect.], **9** [7] 42 (1940).—The brochure is recommended to anyone having contact with lead or the control of lead poisoning, either from the medical or the engineering point of view. K.R.

PATENT

Preparation of roofing granules, etc. J. J. STANKO (American Mineral Co.). U. S. 2,242,443, May 20, 1941 (Oct. 18, 1938).

HOW AN ISSUE OF CERAMIC ABSTRACTS IS PREPARED

Ceramics is a world-wide industry. Technological and scientific literature of importance to American ceramists appears in many languages. How is this material collected from all parts of the world, condensed, and published in the monthly issues of *Ceramic Abstracts* by the American Ceramic Society?

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